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RAY T. BAYLESS, *Editor*

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No. 9

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X-RAY LINE BROADNESS OF METALS AND ALLOYS AND ITS RELATION TO HIGH TEMPERATURE STABILITY

BY J. E. WILSON AND L. THOMASSEN

Abstract

Recovery of X-ray line sharpness in cold-worked metals and alloys is studied as a function of time and temperature of annealing. Line sharpening is caused by diffusion of displaced atoms to normal lattice positions; in the case of nickel at low annealing temperatures, the process involves a long induction period. A quantitative time-temperature relationship for line sharpening is determined for nickel, copper and the alpha brasses. A mechanism of recovery, based on correlations with hardness, electrical conductivity and other data, is described.

Line sharpening temperatures may be used to predict immunity from "season cracking" in brasses.

Creep data on brasses, copper-nickel alloys and a series of pearlitic manganese and manganese-molybdenum steels are correlated with line broadness data. In a continuous series of solid solutions, the alloy of maximum line-sharpening temperature has the optimum creep characteristics. The poor creep strength of 60-40 brass above 500 degrees Fahr. is due to inferior temperature stability of the beta phase. A secondary maximum in creep strength at 900 degrees Fahr. in one of the manganese-molybdenum steels is paralleled by a maximum in a precipitation hardening effect which is disclosed by X-ray and hardness measurements.

INDUSTRIAL requirements for materials of good high temperature properties have increased enormously in the past decade. The outstanding qualifications for such service are high limiting creep stresses and resistance to corrosion and oxidation under operating conditions. The behavior of metals and alloys at high temper-

This paper is based on a dissertation submitted by J. E. Wilson in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the University of Michigan.

Of the authors, J. E. Wilson is metallurgist for the Kelvinator Corp., Detroit, and L. Thomassen is assistant professor of Chemical Engineering, University of Michigan, Ann Arbor, Mich. Manuscript received August 9, 1934.

atures has therefore become very important to the metallurgist, and probably will be for years to come.

The most direct way of investigating the high temperature properties of an alloy is to subject it to tests that simulate operating conditions as nearly as possible or convenient. Tests of this sort are necessarily time consuming and limited in applicability to the particular alloy in question.

Creep is essentially plastic deformation which occurs at such a temperature that the strain-hardening is partially offset by the isothermal softening or recovery. The equilibrium creep rate at a given temperature and stress is dependent on the difference between rates of strain-hardening and recovery and on the inherent cohesion properties of the particular metal or alloy under observation. Examination of this problem from the point of view of the general behavior of cold-worked metals and alloys on annealing might accordingly be expected to yield valuable correlations. Among the properties which might be studied are hardness, electrical conductivity, magnetic induction, and X-ray diffraction effects. The last named property has served most extensively as a tool in the present investigation.

In 1925 van Arkel (1)* reported that the X-ray diffraction lines produced by the powder method (Hull-Debye-Scherrer method) were made broadened and diffuse for a number of cold-worked metals, and that this effect disappeared on annealing. Similar observations were reported by Davey (2) on tungsten powders.

This effect has since been investigated somewhat more thoroughly, but the reported experiments are not all in agreement. It is generally accepted that the broadening is due to internal strains rather than extremely fine grain size. (3, 4, 5) There is, however, a wide spread of opinion as to the role this type of lattice distortion plays in the other observed effects of plastic deformation such as hardening and decrease of electrical conductivity. One group of investigators (4, 5) has advanced the hypothesis that the lattice distortion revealed as X-ray line broadening is responsible for the hardening of metals on cold-working based on an alleged parallelism between the increase of hardness and the line broadness with the degree of deformation.

Others (3) believe that the broadening is a result of a special kind of lattice distortion and is more or less independent of strain-

*The figures appearing in parentheses refer to the appended bibliography.

hardening. This belief is based on the existence of strain-hardening in certain metals, such as aluminum, which do not show appreciable broadening of X-ray lines after deformation at room temperature.

Published data and theory concerning the recovery of line sharpness on annealing are not in good agreement. Dehlinger (3, 6) believes that the sharpening temperature coincides with the recrystallization temperature, while Sekito (7) thinks that line broadness decreases gradually with increased annealing temperature. Burgers and van Arkel (8) maintain that for any annealing temperature below the recrystallization temperature the broadness diminishes quickly to a constant end value, and that a fixed unsharpness remains for each annealing temperature. These conclusions, however, were based only on experiments with tungsten wires.

MATERIALS INVESTIGATED

This investigation falls in two parts. In the first part, pure metals were investigated; in the second, alloys, on which creep test data were available, were studied. In order to eliminate anomalous behavior due to impurities, an effort was made to obtain as pure samples as possible for the first part of the investigation. For the second part, we were also fortunate in obtaining samples of brass and of an unusual series of pearlitic manganese steels from the Department of Engineering Research of this University, through Dr. C. L. Clark, who also furnished us with samples of Admic and Monel metal. All of these samples have been investigated by Clark and White so that very complete creep data were available.

EXPERIMENTAL TECHNIQUE

In order to eliminate the degree of deformation as a variable, all the pure metals were worked as severely as possible, that is, to incipient failure, while in the alloys, the degree of deformation was kept as constant as possible throughout each series. Except for certain special experiments, all of the cold-working was done by means of a belt hammer at room temperature, care being taken to dip the small specimens in water between the blows of the hammer to prevent any appreciable heating.

The strips of work-hardened material were then cut into specimens one-half inch square, numbered, ground and polished. After the polishing operation, the samples were given the desired annealing

treatment in an electric furnace controlled by a potentiometer controller. To further decrease the temperature variations, specimens were inserted in a cast iron annealing block. Temperatures were read with a portable potentiometer which was calibrated against a precision potentiometer. The chromel-alumel couple used had been calibrated against the melting point of pure zinc. Its readings were

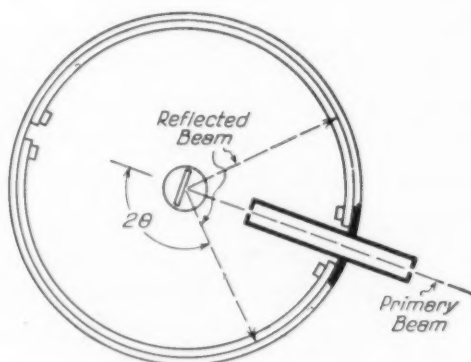


Fig. 1—Sketch of Debye Camera.

also compared to those of a calibrated mercury thermometer and a proper correction curve worked out.

The time of annealing in this report is the designated period after the specimen has come to the desired temperature. In all cases the furnace was at temperature when the specimens were inserted. The time required for specimens to reach the desired temperature was found to lie between two and four minutes. After annealing, the samples were severely deep-etched to relieve any surface strains not representative of the body of the metal.

X-ray diagrams were then taken of the samples by placing the prepared surface parallel to the axis of a cylindrical camera so that the axis was in the plane of the surface. The diameter of the cameras used was 205 millimeters and the beam of X-rays striking the surface at a 90-degree angle was diffracted from the surface of the sample to the cylindrical film. This "back reflection" method has the advantage that the lines of largest angle of diffraction will be present, thus giving the maximum resolution of the $K\alpha$ doublet, so that the breadth determinations can be made on a single line. A sketch of the camera is shown in Fig. 1.

In Fig. 2 is shown typical diagrams of a cold-worked and of an annealed specimen of brass. The radiation used was the Fe-K radiation generated in a demountable X-ray tube of the Siegbahn-Hadding

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type. The exposure times were between 30-40 milliamperes-hours at 20 KV peak.

Broadness of the lines was determined by use of the large model Moll microphotometer. The results are expressed in terms of the breadth of the $K\alpha_1$ line at one-half its maximum blackening. Photom-

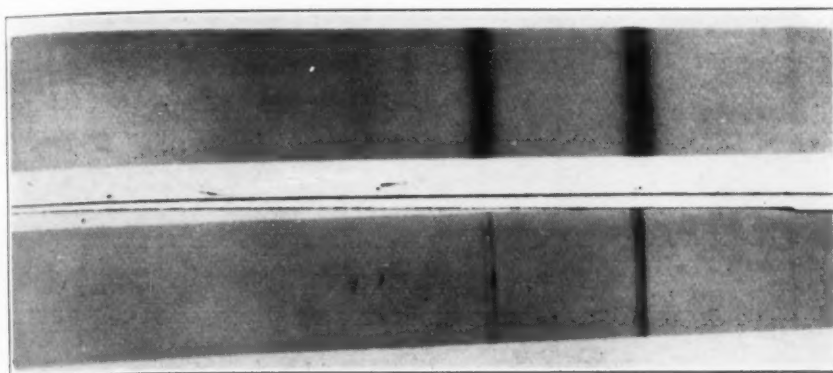


Fig. 2—"Back-Reflection" X-Ray Diagrams; Upper Diagram Shows (222) and (113) K Lines of Cold-Worked 70-30 Brass, While Lower One Shows Same Lines in Specimen Annealed at 280 Degrees Cent.

eter magnification is 7 times except where otherwise noted. Fig. 3 shows typical photometer traces. A typical series of measurements are given in Table I.

RESULTS

Pure Metals

Aluminum—Van Arkel (1) and Dehlinger (3) in their work on broadening of X-ray lines on cold working of metals report that the lines of several of the soft metals including lead, tin, zinc and aluminum were not broadened by cold work. On the basis of these results, Dehlinger has proposed a different mechanism of cold working for aluminum than for other metals.

Considering the low melting points of these metals, it may be probable that there is a connection between these and the temperature of working of the metal which presumably is room temperature. It was, therefore, decided to determine if aluminum could be made to fall in line with the other metals by working it at a lower temperature. A cold drawn piece of this metal was therefore annealed for three hours at 250 degrees Cent. (480 degrees Fahr.). Specimens were then compressed in a universal testing machine, some at room temperature and some at -76 degrees Cent. (-105 degrees Fahr.). In

the latter operation the samples were immersed in acetone, cooled by dry ice, for fifteen minutes before the compression began. The compressed specimen was transferred to an X-ray camera and the

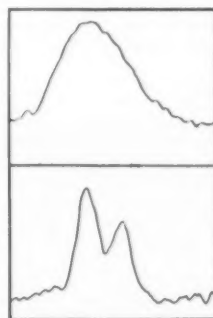


Fig. 3—Moll Microphotometer Traces for Cold-Worked and Annealed Brasses.

exposure completed within two hours. The measurements of the photometric records are shown in Table I.

Table I
Measurements of Photometric Records

Treatment	$\frac{1}{2}$ Value Line Broadness (28X) Millimeters
Annealed	22.2
Worked at 25 degrees Cent. (77 degrees Fahr.)	23.6
Worked at -76 degrees Cent. (-105 degrees Fahr.)	35.8

As will be seen, the sample worked at the lower temperature has an appreciable increase in line broadness.

Copper—Cylindrical specimens of annealed copper were compressed at the desired temperatures under a load of 3000 kilograms, cooled quickly in air, etched, and X-ray pictures prepared. The results shown in Table II indicate that copper, as well as aluminum, may be worked at such a temperature that no line broadening will result.

Table II
Measurements of Photometric Records of Copper Specimens

Deformation Temperature	$\frac{1}{2}$ Value Breadth* (113) $K\alpha_1$
25 degrees Cent. (77 degrees Fahr.)	16.4 millimeters
150 degrees Cent. (300 degrees Fahr.)	18.0 millimeters
260 degrees Cent. (500 degrees Fahr.)	21.4 millimeters
400 degrees Cent. (750 degrees Fahr.)	19.2 millimeters
510 degrees Cent. (950 degrees Fahr.)	10.7 millimeters

*Microphotometer magnification is $\times 28$.

In addition to these experiments, the material was cold-forged in the standard manner to a reduction of thickness of 90 per cent, then annealed for 1-hour periods at selected temperatures. Broadness of the (113) line is plotted against annealing temperature in Fig. 4. The curve indicates the rapid recovery within a narrow temperature range; in this case it is less than 40 degrees Cent. (105

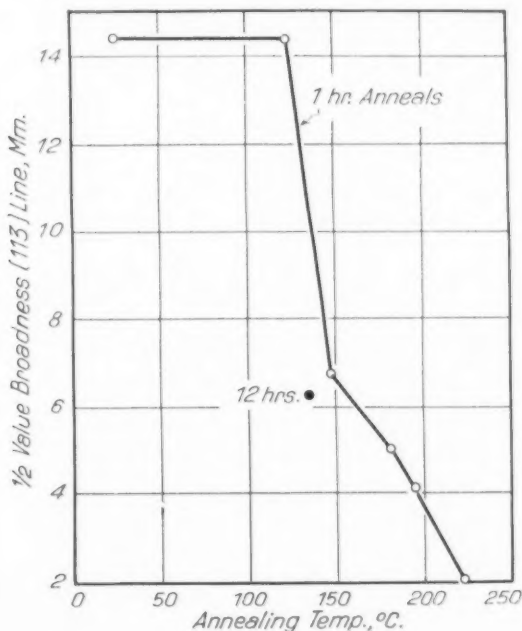


Fig. 4—Recovery of High Purity Copper from Line Broadness.

degrees Fahr.). At annealing temperatures above 150 degrees Cent., (300 degrees Fahr.) individual sharp spots appear in the continuous powder lines, indicating that gross recrystallization is beginning.

Nickel—The material was cold-forged to a reduction of thickness of 75 per cent after annealing one hour at 700 degrees Cent. (1290 degrees Fahr.), then tested according to the standard procedure. The results of the line broadness measurements on the (113) lines are reproduced in Figs. 5, 6, and 7. The annealing treatments were planned so as to study the effect on line broadness of the two variables present, namely, time and temperature, keeping first the one, then the other, constant, and finally investigating the course of line sharpening for various annealing periods.

The amount of sharpening produced by various heating times at 512 degrees Cent. (955 degrees Fahr.) is shown in Fig. 5. There

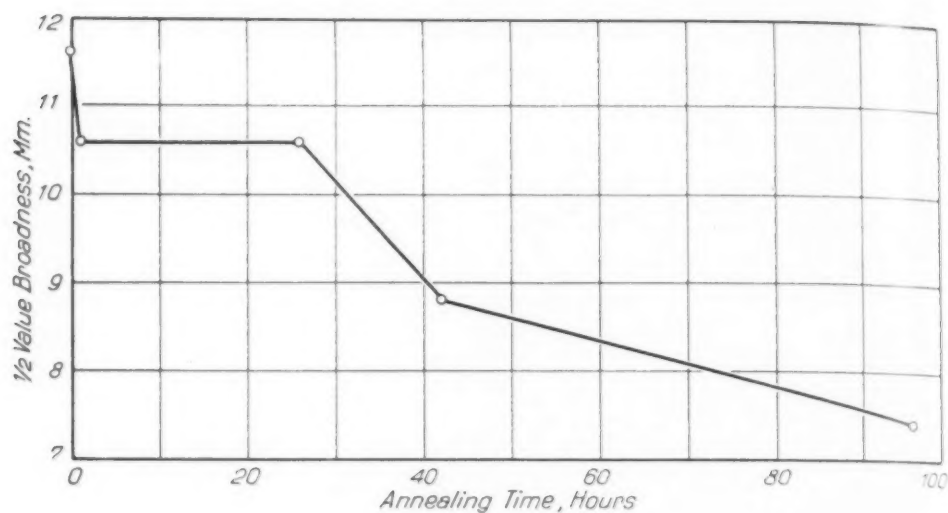


Fig. 5—Effect of Time at 512 Degrees Cent. on Width of (113) Line of Nickel.

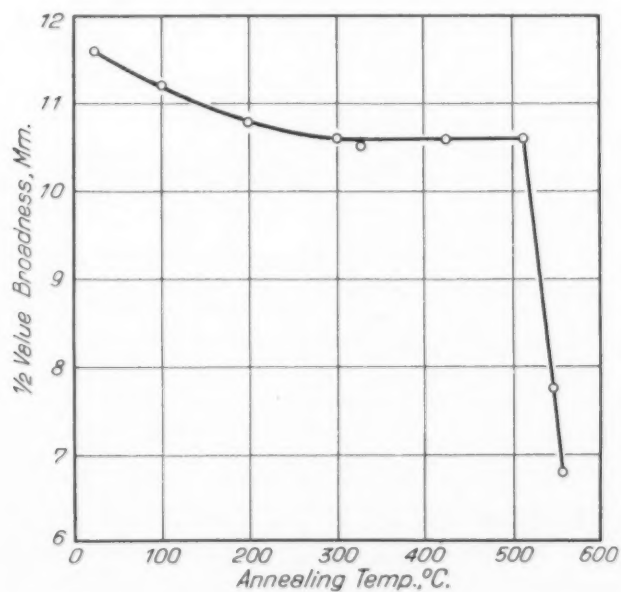


Fig. 6—Effect of One Hour Anneals at Various Temperatures on Broadness of (113) Line of Cold-Worked Nickel.

is almost no change from 1 hour to 26 hours, and relatively rapid sharpening takes place between 26 and 42 hours. Then a slow sharpening occurs on further heating.

The effect of 1-hour anneals at various temperatures is shown in Fig. 6. The line broadness shows a slight but definite drop in the interval 25 to 300 degrees Cent. (77-570 degrees Fahr.), and then

remains constant to 512 degrees Cent. (955 degrees Fahr.). In the temperature interval 512 to 564 degrees Cent. (955-1050 degrees Fahr.) the drop is sharp, changing from 10.6 at the former to 6.8 at the latter temperature. At 564 degrees Cent. (1050 degrees Fahr.) distinct resolution of the doublet was observed so the anneals were stopped.

The course of line sharpening for various annealing periods is shown in Fig. 7. The specimens were annealed for various lengths

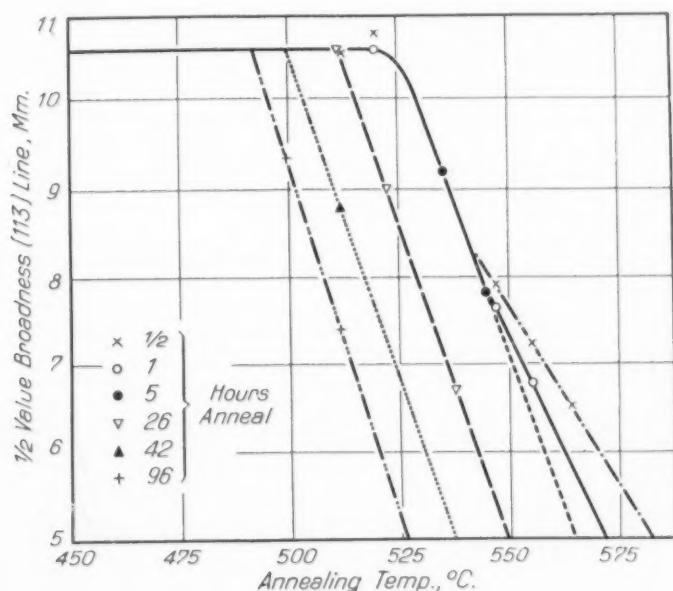


Fig. 7—Recovery of Line-Sharpness on Annealing Cold-Worked Grade A Nickel.

of time at chosen temperatures and the results seem to show that the recovery of line sharpness takes place over a broader temperature range in short-time than in long-time anneals. More particularly, Fig. 7 shows that there is little difference in slope of recovery curves for anneals of 5 hours or longer and that the slope becomes progressively flatter for the 1-hour and 30-minute series.

In order to reduce these data to a common basis, the individual curves have been extrapolated to half-value widths of 5.0. This value was chosen because it corresponds to the lowest broadness value attained with this metal without introducing complications due to recrystallization. The temperatures at this half-value have been plotted against the logarithms of the corresponding annealing time in Fig. 8. The straight line shown was considered the best fit for the data.

Iron—Specimens of cold-worked Armco iron were annealed for 1 hour and X-ray diagrams prepared. Broadness measurements were made on the (220) and (211) lines, with results as reproduced in Fig. 9. It is noteworthy that although the amount of broadening is greater for the higher order lines, in confirmation of experimental

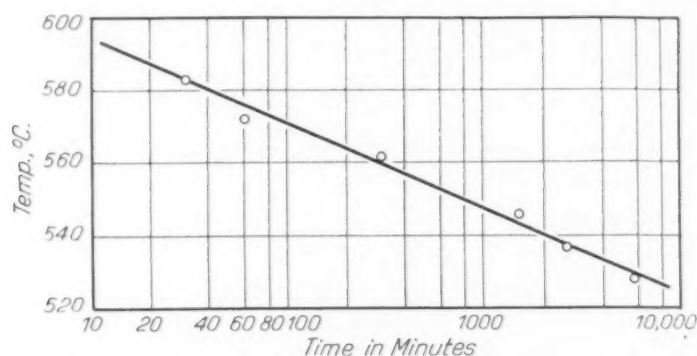


Fig. 8—Relation Between Time and Temperature of Annealing Grade A Nickel to Attain Constant Line-Sharpness of 5.0.

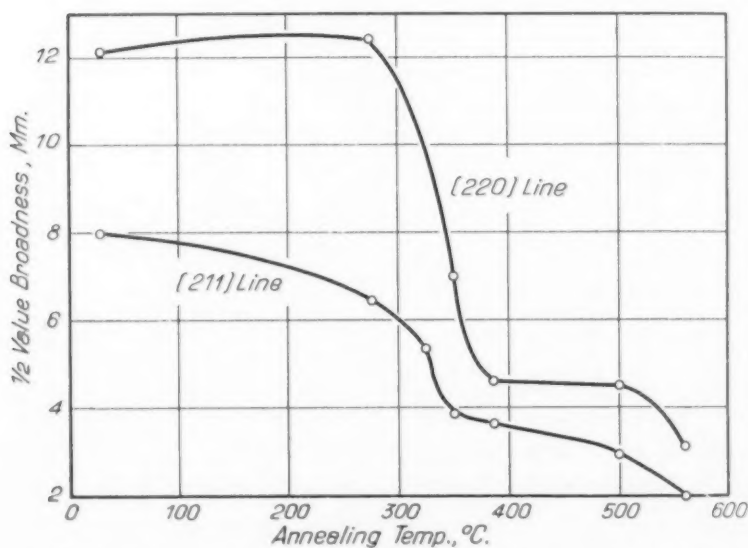


Fig. 9—Recovery of Armco Iron from Line Broadness. One Hour Anneals.

results of Wood (9) and of Caglioti and Sachs (10), the temperature of inflection points of the recovery curves are independent of the order of line. The dormant period in line sharpening from 400-500 degrees Cent. (750-930 degrees Fahr.) is followed by a secondary sharpening which corresponds to the formation of individual sharp spots on the lines which correspond to gross recrystallization grains, rather than the general recovery from line broadness.

Summary of Experimental Results for Pure Metals

- (1) The temperature of working affects the amounts of broadening attainable as shown by copper and aluminum.
- (2) In the case of iron, the amount of broadening is greater for higher order lines but the course of line sharpening is essentially the same; i.e., the inflection points of the recovery curves occur at the same temperature for both sets of lines.
- (3) The temperature range of line sharpening becomes progressively greater for annealing times less than 5 hours in the case of nickel.
- (4) The time for a constant recovery effect as shown by constant line broadness is a logarithmic function of the annealing temperature of nickel.

COPPER-ZINC ALLOYS

Three brasses of wide commercial application were tested—namely, the 85-15, 70-30 and 60-40 alloys. The compositions are given in Table III.

Table III
Chemical Compositions of Selected Copper-Zinc Alloys

Designation	Copper Per Cent	Zinc Per Cent
R-85	85.00	14.92
B	69.48	30.44
E-1*	60.21	39.72

*Contains 0.16 per cent tin.

The annealed alloys were cold-forged until they cracked. This occurred at a reduction of between 85 and 90 per cent. The samples were annealed in the usual way. Broadness measurements were obtained on the (222) line. Hardness measurements were made on these specimens and plotted along with some of the line sharpening curves.

85-15 Brass—From Fig. 10 it will be seen that the major portion of the sharpening has been accomplished at about 265 degrees Cent. (510 degrees Fahr.) for the 1-hour anneals, at which temperature the hardness has just begun to drop. The drop in broadness for this annealing time and for ten minutes, twelve hours and 107 hours is shown in Fig. 11, with a more extended temperature axis. The tendency here is, as in the case of pure nickel, that there is a larger temperature range of sharpening for short-time than for

long-time anneals. Sharp spots were not noticed on the lines from specimens which had been annealed for 1 hour at temperatures less than 300 degrees Cent. (570 degrees Fahr.), indicating that gross recrystallization involving grain growth does not occur at much lower temperatures. The flat foot portion of the recovery curve corresponds to the inception of softening and gross recrystallization.

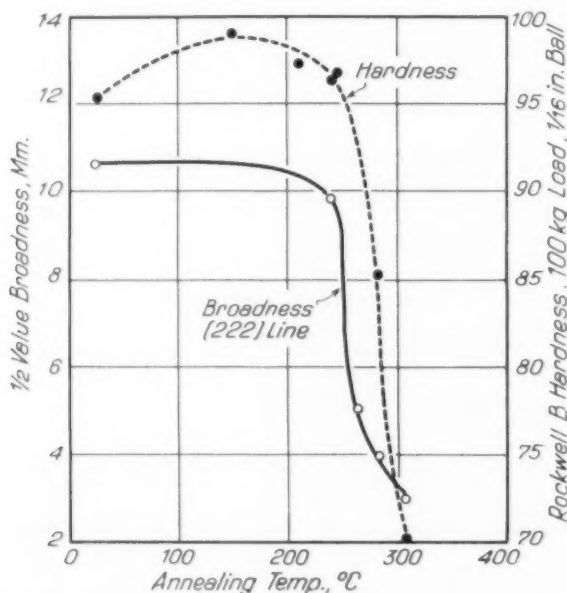


Fig. 10—Recovery of Cold-Worked 85-15 Brass, Annealed for 1 Hour.

70-30 Brass—Fig. 12 indicates sharpening of lines and recovery from work hardening in the 70-30 composition. The characteristics of these curves are the minor increase in hardness of brass in intermediate temperatures, and the extreme rapid drop in line broadness apparent in the temperature range 245-255 degrees Cent. (475-490 degrees Fahr.). In this range, about 90 per cent of the sharpness is restored, while the recovery from work hardness amounts to only 20 per cent.

The curves for the recovery of line sharpness with varying annealing times are reproduced in Fig. 13. These are the results of a limited number of experiments, but they seem to prove that rate of sharpening with respect to temperature is constant for annealing times of one hour or more.

60-40 Brass—This alloy is composed of two solid solution phases of different lattice structure. In order to study the recovery of this alloy from the effects of cold work, both the α and the β phases were

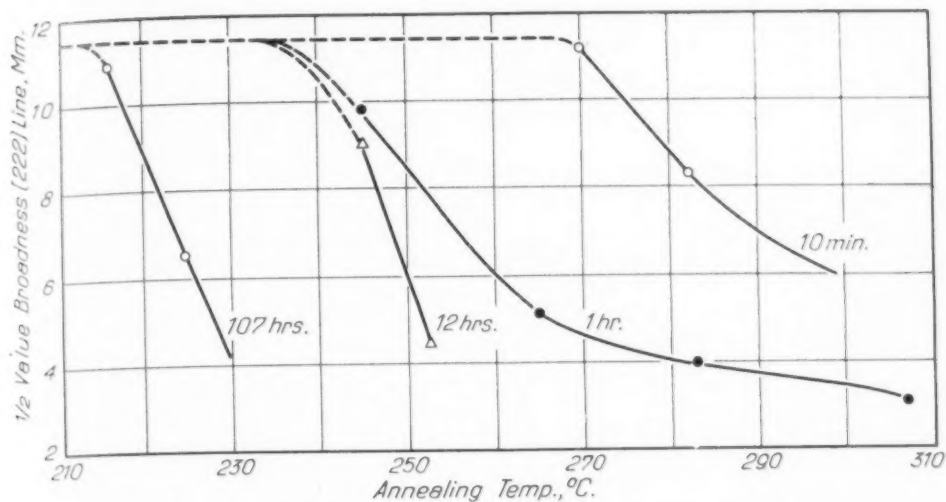


Fig. 11—Recovery of Line Sharpness on Annealing Cold-Worked 85-15 Brass.

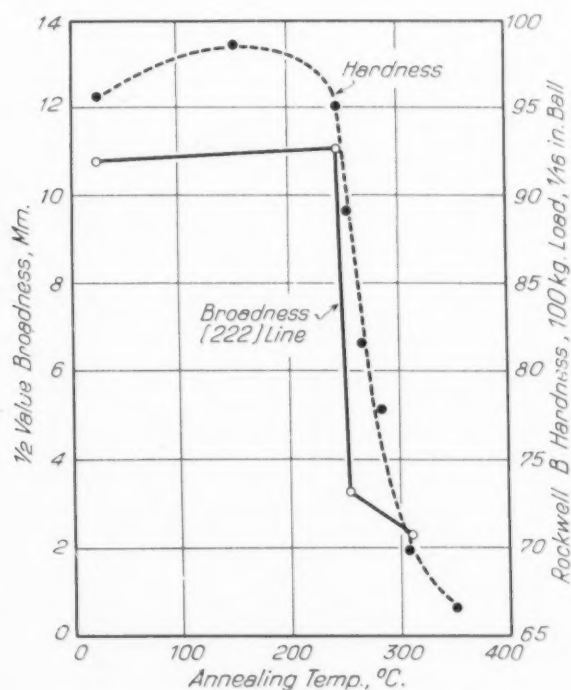


Fig. 12—Recovery of Cold-Worked 70-30 Brass, Annealed for One Hour.

investigated. It is easily seen that the X-ray method furnishes an ideally simple method of attaining this goal. Fig. 14 shows the recovery of line sharpness for the (222) and the (220) lines from the α and the β phases, respectively, together with the Rockwell hardness for specimens annealed 1 hour at the indicated temperatures. These

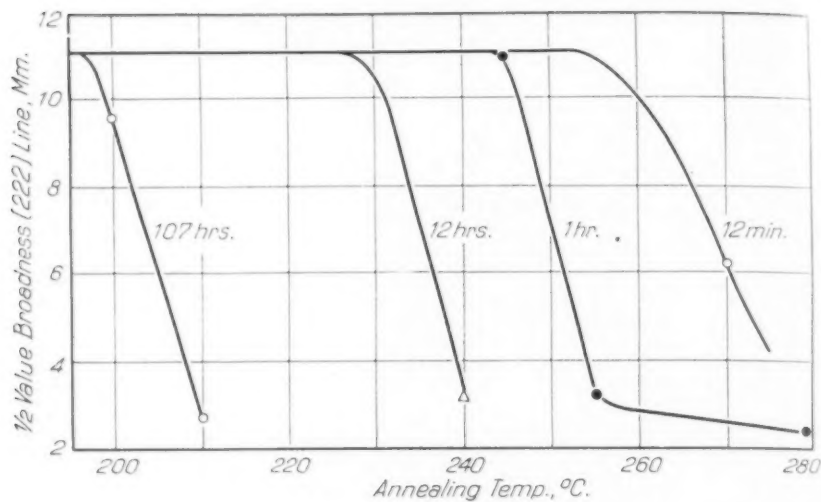


Fig. 13—Recovery of Cold-Worked 70-30 Brass From Line Broadness.

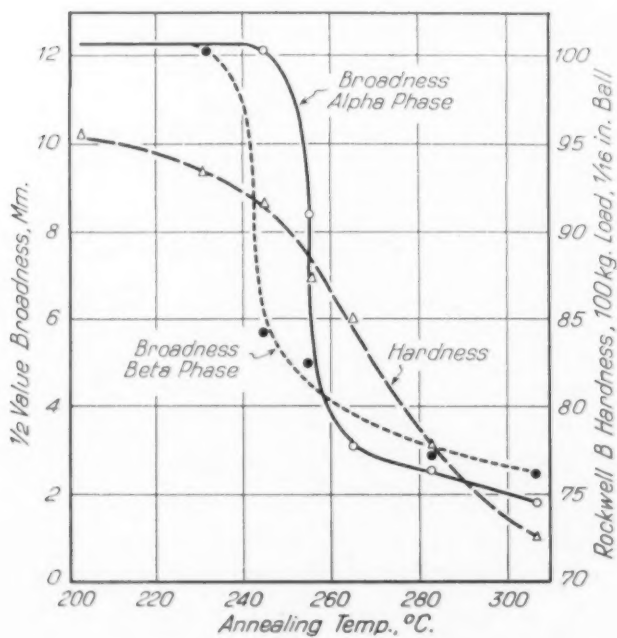


Fig. 14—Recovery of Cold-Worked 60-40 Brass. One Hour Anneals.

curves clearly show that the recovery from the effects of cold work has its inception in the β phase at a lower temperature than in the α phase. The hardness recovery is apparently an average effect of the two phases as would be expected from the nature of the test.

ADNIC AND MONEL METAL

These alloys were chosen as representative copper-nickel alloys of commercial importance. The chemical analysis of the samples used are shown in Table IV.

Table IV
Composition of Adnic and Monel Metal

	Copper	Nickel	Iron	Tin
	Per Cent	Per Cent	Per Cent	(Nominal)
Adnic	70	29	...	1
Monel	29.7	67.7	1.77	(Mn) 1.28

The presence of iron, manganese and tin prevent their being true copper-nickel alloys but it was felt that the results obtained would be of more general interest than those found on synthetic alloys of high purity.

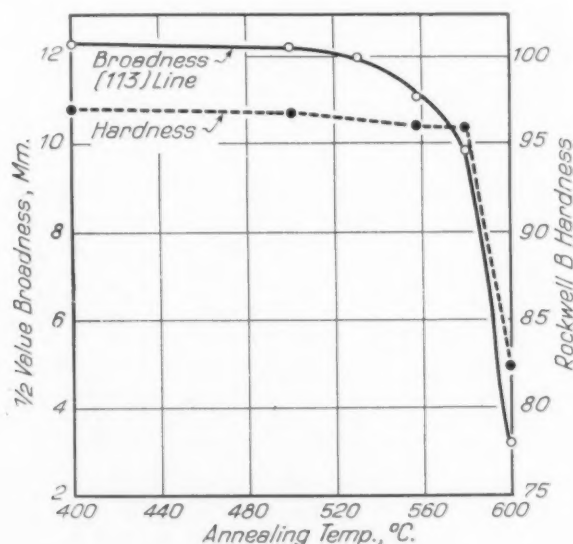


Fig. 15—Recovery of Monel Metal. One Hour Anneals.

The experiments with these alloys were limited to 1-hour anneals, and as Figs. 15 and 16 show, the recovery takes place over a rather broad temperature range in both alloys. The region of rapid sharpening is between 575 and 600 degrees Cent. (1065-1110 degrees Fahr.) for the Monel. In the case of Adnic, the recovery is apparently a 2-stage process with drops occurring between 400 and 500 degrees Cent. (750-930 degrees Fahr.) and again between 560 and 580 degrees Cent. (1040-1075 degrees Fahr.).

PEARLITIC STEELS

The alloyed steels which were made the subject of this investigation include a series of carbon-manganese steels ranging from 1.62 to 2.67 per cent manganese, a series of carbon-manganese steels varying between 1.25 to 3 per cent manganese with constant additions of

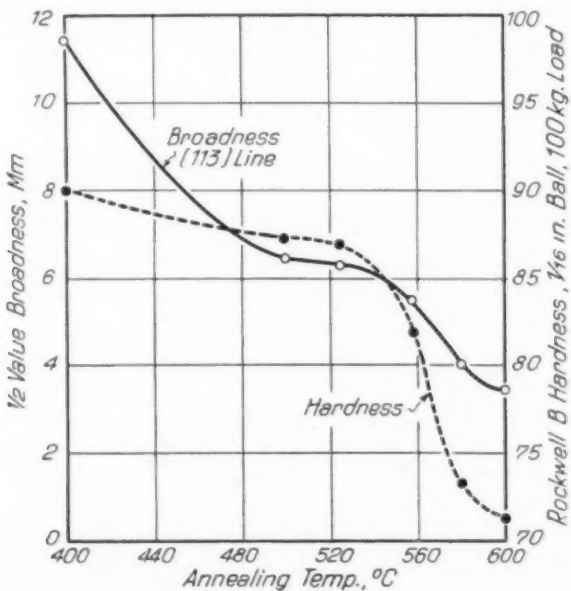


Fig. 16—Recovery of Cold-Worked Adnic. One Hour Anneals.

about 0.25 per cent molybdenum, and a carbon-manganese-molybdenum steel modified by the addition of vanadium. The chemical analyses are given in Table V. The amount of carbon present is about 0.16 per cent and nearly constant for all steels.

Table V
Chemical Compositions of Selected Alloyed Steels

Designation	C	Mn	Mo	V Per Cent	Si	P	S
MM-5	0.14	1.62	0.15	0.019	0.017
MM-6	0.16	2.10	0.22	0.020	0.024
MM-7	0.18	2.67	0.10	0.018	0.028
MM-10	0.15	1.10	0.20	0.36	0.02	0.018
MM-8	0.15	1.25	0.25	0.19	0.028	0.018
MM-1	0.17	1.40	0.22	0.33	0.018	0.020
MM-9	0.15	1.75	0.25	0.15	0.017	0.024
MM-2	0.15	2.06	0.21	0.19	0.019	0.019
MM-3	0.17	2.52	0.26	0.15	0.020	0.030
MM-4	0.16	2.99	0.24	0.23	0.024	0.017
QV	0.18	2.00	0.32	0.20	0.23	0.016	0.025

The preparation of the samples was as follows: Bars of 1-inch diameter were annealed above their critical temperatures and furnace-

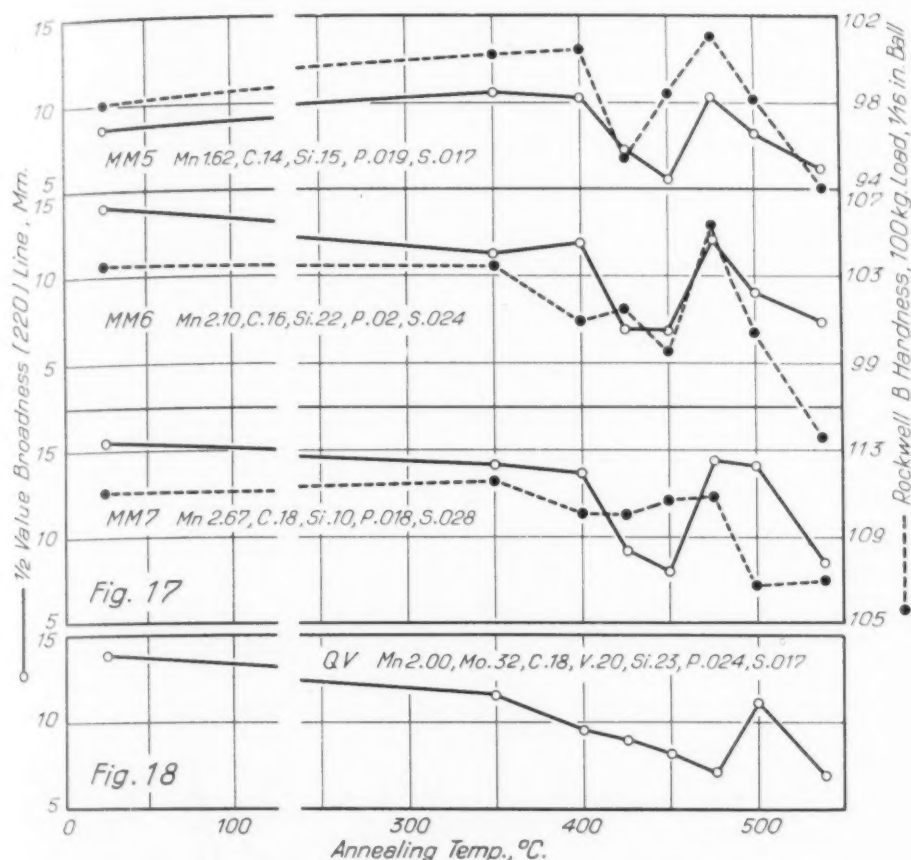


Fig. 17—Hardness and Sharpness of (220) Lines of Cold-Worked Manganese Steels After 2-Hour Anneals at Indicated Temperatures.

Fig. 18—Sharpness of (220) Lines of Cold-Worked QV Steel After 2-Hour Anneals at Indicated Temperatures.

cooled, and then reduced in a shaper to flats of $\frac{3}{8}$ -inch thickness. The flats were cold-forged to $\frac{1}{4}$ inch thickness, thus giving a plastic deformation of 33 per cent on the original thickness. The specimens were then treated as before, except that the temperature was controlled to only plus or minus 6 degrees Cent. After the specimens had been heated 2 hours at the desired temperature, the current was shut off, and the samples allowed to cool in the furnace. To get comparable results, samples of each composition were assembled in sets, and annealed simultaneously at the designated temperature.

In Figs. 17 to 19 are graphically summarized the X-ray and hardness data obtained on these series of steels. The curves show a general parallelism between broadness and hardness measurements, but the recovery is more complex, due apparently to a hardening of the precipitation type. The significance of the individual curves will be discussed later.

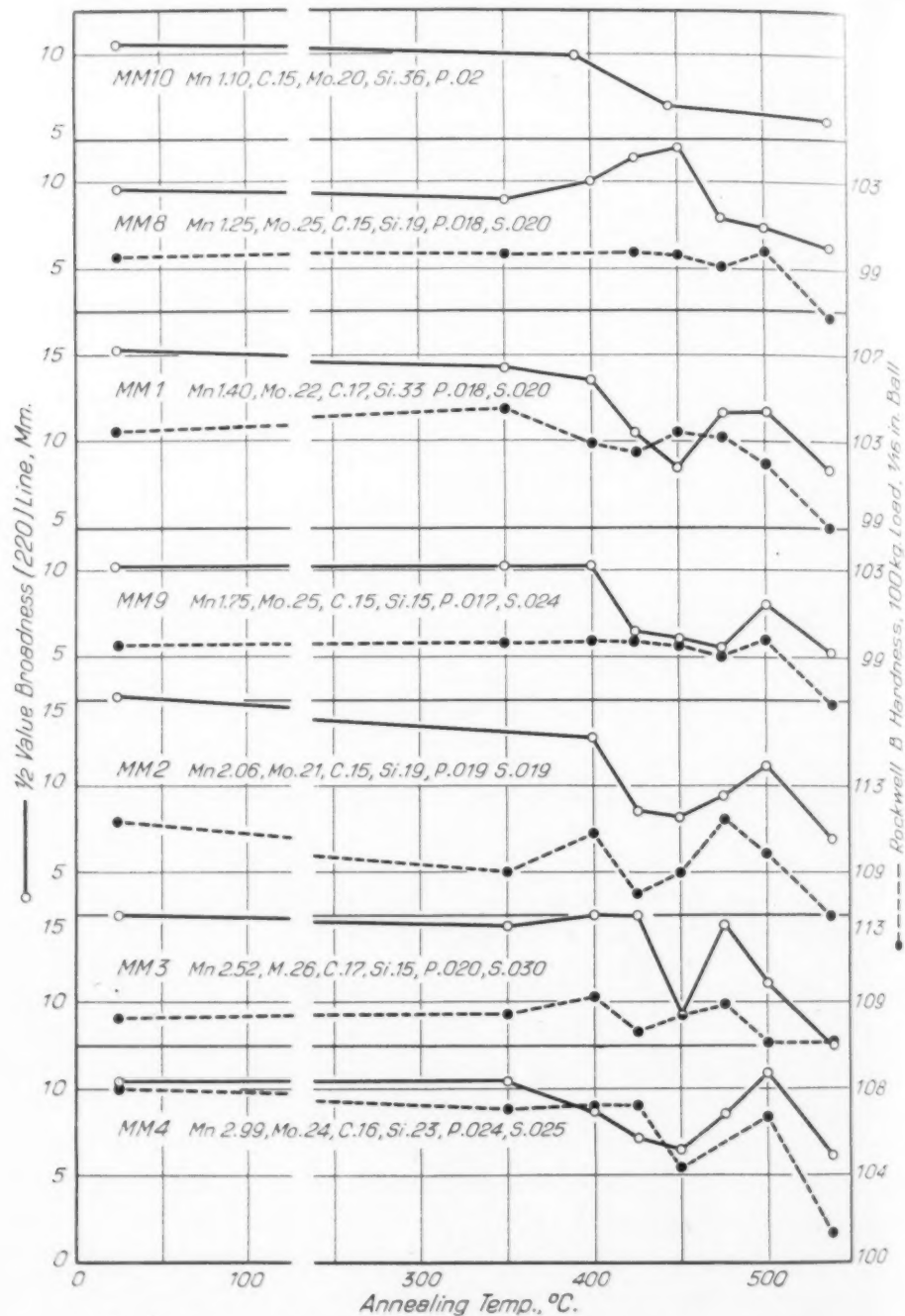


Fig. 19—Sharpening of (220) Lines of Cold-Worked Manganese-Molybdenum Steels on Annealing Two Hours at Indicated Temperatures.

INTERPRETATION OF RESULTS

These results, when considered in their relation to other known phenomena of the recovery of metals and alloys from the effects of

cold-work, lead to a conception of the mechanism of plastic deformation and recovery which is in many ways similar to previously advanced hypotheses but which requires modification of certain accepted concepts for agreement with the assembled data.

Data from the literature on such effects of plastic deformation as have been noted by electrical, magnetic and hardness measurements are included in the following discussion. The mechanism of plastic deformation and recovery obtained by these correlations is applied particularly to the questions of season cracking in brass and to the creep properties of metals at elevated temperatures. It is shown that the X-ray data give good agreement with accelerated season cracking tests and the creep data on the alloys studied in this investigation.

PLASTIC DEFORMATION OF ALUMINUM AND COPPER

It was shown in the experimental part that aluminum can be worked at such a temperature that line broadening results, and that, on the other hand, copper can be worked at such a temperature that the lines remain sharp although the converse is true at room temperature in each case. Dehlinger (3) advanced the hypothesis that metals like aluminum, zinc, tin and lead do not exhibit line broadening on cold-working (presumably at room temperature) because the lattice is bent or crumpled sharply in some parts with long intervals of undistorted lattice between. This type of crumpling reduces the probability of line broadening, according to the Laue conception of the space lattice. Dehlinger believes this type of deformation is inherent to such metals; however, he did not discuss the effect of deformation temperature. Expressed as a ratio of deformation temperature to melting point in absolute Centigrade units, room temperature or "cold" working is performed at 0.32 for aluminum, and at 0.22 for copper. By reducing this ratio to 0.24, broadening was produced in aluminum. This indicates that a change in the nature of lattice crumplings is in some way connected with the ratio of deformation to melting temperature of the material.

There are, however, differences in the plastic deformation of aluminum and copper which may be ascribed only to differences in atomic structure associated with their positions in periodic classification of the elements. Thus, aluminum retains hardness due to working at room temperature for an indefinite period; whereas, the data of Sauerwald and Globig (11) indicate that copper worked at tem-

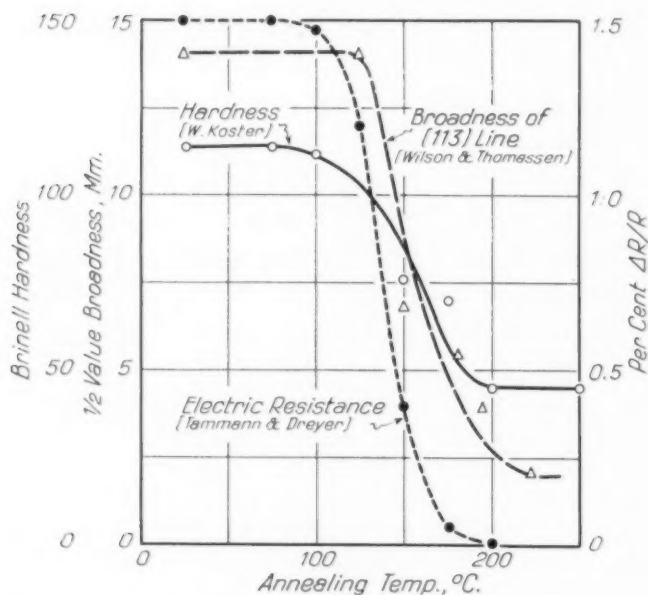


Fig. 20—Recovery of Cold-Worked Copper.

peratures which do not result in line broadening will lose its work hardness in a relatively short time. This is also brought out by the fact that the temperature at which no broadening occurs in pure copper is 0.55 when expressed as a ratio to the melting point compared to 0.32 for aluminum at room temperature.

Recovery of Pure Metals from Effects of Cold Work

Such differences as were pointed out between aluminum and copper also become evident on examination of data on the cold-working and annealing of copper, iron, and nickel. The ways in which differences in atomic structure affect the deformation and recovery characteristics are clarified by a study of the line broadness and hardness data in conjunction with results available in the literature on electrical conductivity and magnetic induction as well as certain other effects.

Recovery of Copper—It was suggested in discussing the plastic deformation experiments that differences in behavior of copper and aluminum were due to differences in atomic structure. Some of the salient features of the recovery of copper are reproduced in Fig. 20. The hardness data are taken from the work of Koster (12) for a degree of cold work corresponding to that used in the X-ray experiments. He found, however, that the inflection point of hardness

recovery curve varied between 90 and 390 degrees Cent. (95-735 degrees Fahr.) as the degree of reduction decreased from 93 to 3 per cent, thus showing a large dependence of recovery temperature on the degree of deformation.

The curves show that the recovery of line sharpness, hardness and electrical conductivity all occur at almost identical temperatures. This coincidence has been used by certain investigators as evidence that the type of atom distortion responsible for strain hardening is also the cause of electrical resistance changes. If this is true, the effects should also be parallel in their increase during plastic deformation. However, according to Tammann and Dreyer (13), the hardness increases rapidly with lower degrees of deformation, the curve becoming almost asymptotic to the degree of deformation axis between 90 and 98 per cent reduction, while the increase of electrical resistance is very slow up to 60 per cent reduction, it then increases rapidly as reductions approach 100 per cent.

W. A. Wood (4), and Caglioti and Sachs (10) both found that the line broadness of copper increases in a manner which is somewhat similar to the hardness, in that it increases more rapidly with lower degrees of deformation. However, Caglioti and Sachs were unable to show complete parallelism for these effects and their calculations indicate that energy must be stored in the lattice in some form other than that evident as line broadening.

The change in electrical conductivity of copper on cold-working cannot in the face of these data be ascribed to the same type of lattice distortion which causes either the hardening or the line broadness. Consideration of the above facts has suggested the following explanation of the mechanism by which the various phenomena of cold-working and recovery of copper occur.

As deformation progresses, in addition to slip, there occurs a displacement of atoms within the extent of each atom block comprising a slip lamella, probably according to a more or less definite period as pictured by Dehlinger (3). This atomic displacement is partially elastic in nature, particularly with small amounts of strain. However, the forces at grain and slip lamella boundaries are so great that actual deformation of outer shell electrons occurs, with the result that cohesion bonds are changed to a molecular type for such boundary atoms. This concept is in a way similar to the hypothesis of Dean and Gregg (14); the atoms so affected are only boundary atoms and due to their small number affect the electrical conductivity but

little; whereas, the hardness or resistance to further slip is greatly increased. The idea of boundary atom deformation has been advanced previously by Dehlinger, who emphasizes the factor of geometric roughening of slip planes, and accordingly is in agreement with the slip interference theory of Jeffries and Archer (15), in that both ascribe hardening to slip interference caused by roughening of the boundary planes.

Experimental verification of this boundary atom deformation is found in the work of Hengstenberg and Mark (16), who observed that the intensity of reflection for high order lines was markedly reduced in duralumin by the age hardening process, although the breadths of lines were unchanged.

As deformation progresses, the binding of so-called "free" electrons with consequent formation of molecular type cohesion bonds occurs in the interior of atom blocks. This causes decreased electrical conductivity. As an additional factor, the formation of extreme preferred orientations becomes effective at very great degrees of deformation. This factor is probably contributory to decreased electrical conductivity in copper by diminution of electron free path.

On annealing, the bound electrons are freed by input of thermal energy and the unstable molecular bindings change to normal lattice forces and the displaced atoms which cause line broadening return by a diffusion process to normal positions. The sequence of changes which occur on annealing may thus be summarized:

1. Thermal vibration of individual atoms is inhibited by the molecular type bondings. These bonds are decomposed by heat allowing the less stable molecules of the interior of slip lamella to dissociate.
2. This dissociation frees valence electrons and the electrical conductivity is recovered.
3. Thermal vibration of individual atoms occurs in the interior of slip lamella, allowing displaced atoms to diffuse back to normal positions. This results in line sharpening. The relief of internal strains in the interior of atom blocks results in a certain stage of the process in an actual increase in forces on the boundary atoms which causes a condition of abnormal stress at these points, as evidenced by often observed increase in hardness after low temperature anneals.
4. The diffusion of atoms of the body of the lattice block to normal position eliminates the abnormal force components on the atoms of slip planes; the molecular bonds and atom displacements are then eliminated by the further influence of heat and the softening process occurs.
5. Gross recrystallization consists of a diffusion process by

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which two or more lattice blocks of the order of magnitude of 10^{-6} centimeter which have returned to normal condition according to the described processes are joined by a slight change in orientation.

The energy distribution in the cold-worked lattice is Maxwellian in nature, according to evidence of Zickrick and Dean (17) and by van Liempt (18), so the above described processes overlap to an extent which depends on the nature of the metal. In copper the energy

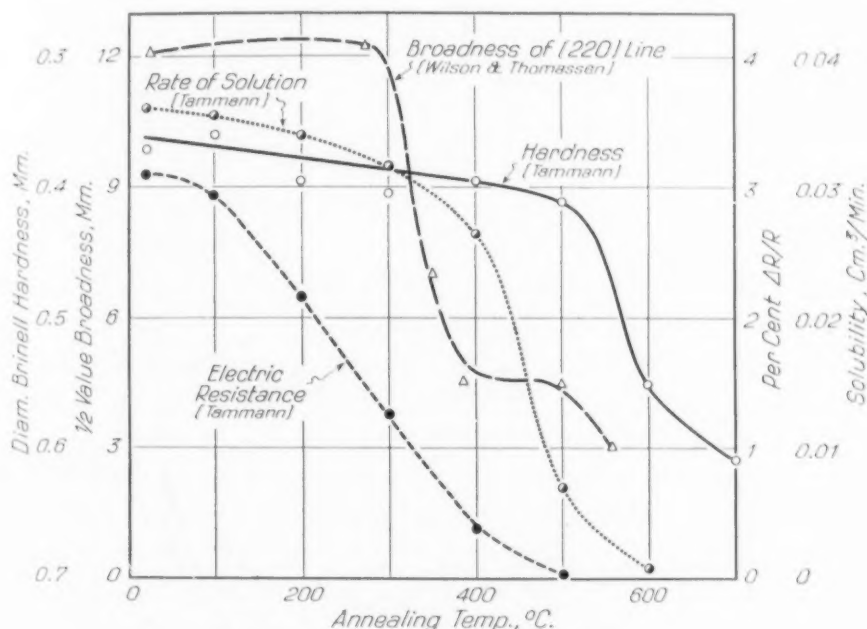


Fig. 21—Recovery of Cold-Worked Iron.

requirements of the decomposition of molecular bonds are apparently only slightly different than those for atomic diffusion and the temperatures for the observed phenomena are accordingly almost identical.

Recovery of Iron—Fig. 21 illustrates the main features of the recovery of iron from the effects of cold work. The data on electrical and magnetic recovery and on rate of solution in reagents are due to Tammann and Moritz (19). These curves show a wide temperature difference for the occurrence of maximum rates of recovery of various properties as shown by the inflection points of the curves. In iron the steps in the recovery process, which are hard to separate in copper, are distinct and easily traced. The inflection point of recovery of electrical conductivity occurs at approximately 250 degrees Cent. (480 degrees Fahr.), and according to the findings of Tammann and Moritz, this point is not affected by the degree of

deformation. The next inflection point, or point of maximum rate of change, occurs in the recovery of line sharpness, which is found to lie at approximately 345 degrees Cent. (655 degrees Fahr.), followed at 430 degrees Cent. (805 degrees Fahr.) by the inflection point of the decrease in rate of solution of iron, and by the corresponding point for hardness drop at 550 degrees Cent. (1020 degrees Fahr.), which coincides with a secondary drop in line broadness between 500 and 600 degrees Cent. (930-1110 degrees Fahr.).

These stages of the annealing process follow very clearly the mechanism outlined in the discussion of the recovery of copper. The iron atoms initially vibrate as molecular units under the influence of heat. This causes the input of sufficient energy for a dissociation process to occur, which results in the observed increase in electrical conductivity. The concept of decreased free path of electrons caused by disordered lattice arrangement is insufficient to explain the decrease in electrical conductivity of iron since if this were the case, the recovery of electrical conductivity should coincide with the elimination of line broadness. The latter effect is induced by the diffusion of individual atoms to normal lattice positions, after vibration of individual atoms is made possible by dissociation of molecular bondings.

The next phenomena in order of temperature is the decrease in rate of solution of iron, which occurs in a temperature interval during which the recovery of line sharpness is dormant. This recovery phenomenon is due to the inception of the dissociation effect in atoms comprising the boundaries of slip units. These are of insufficient volume to affect the electrical conductivity or line broadness to any extent, but the rate of etching is apparently dependent on the condition at the boundaries, since observations with sensitive reagents show initial attack occurs at such points. This boundary distortion is apparently the major factor in the hardening of iron on cold work and in microscopic recrystallization. This is substantiated by the fact that the last stage in the process coincides with the softening period. As outlined under steps 4 and 5 in the discussion on copper, the softening and gross recrystallization stages are conceived as the complete elimination of boundary distortion and movements of the reorganized atom blocks into suitable orientations for reunion into microscopic grains.

Recovery of Nickel—The recovery of nickel is of interest, since this element crystallizes in the same lattice system as copper, while it is comparable to iron in that it is a neighboring ferro-magnetic

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element. Accordingly, it would be expected that the curves for nickel should resemble in some respects both the previously named elements. The curves of Fig. 22 confirm this assumption. The electrical conductivity is recovered at much lower temperatures than are the hardness and magnetic induction effects. In fact, a slight hardening ef-

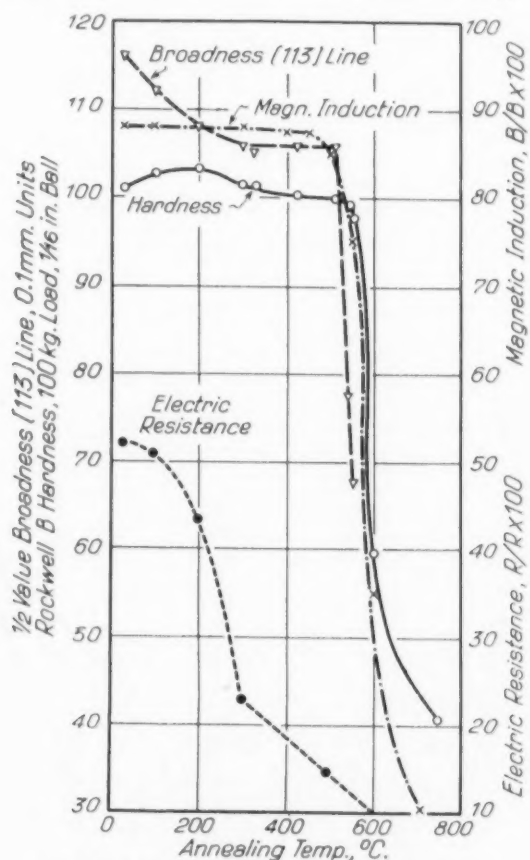


Fig. 22—Recovery of Cold-Worked Nickel.

fect is noted in the temperature range of maximum recovery rate for the conductivity. A slight drop in line broadness is noted in the temperature range 100 to 300 degrees Cent. (210-570 degrees Fahr.), which corresponds to the first stage of electrical recovery.

The difference in temperatures for the main drops in hardness and line broadness is not large but the broadness falls off at definitely lower temperatures than the hardness. For instance, the hardness after a 1-hour anneal at 548 degrees Cent. (1020 degrees Fahr.) is unchanged, while the broadness as measured on the same specimen has decreased from 10.6 to 7.6 on the arbitrary scale of half-value measurements.

It is worthy of note that the magnetic recovery takes place in the same temperature range as the hardness recovery. This fact may be taken to indicate that the magnetic induction phenomena are intimately connected with boundary conditions.

Time-Temperature Law of Recovery of Nickel—In Fig. 8 the temperatures at which nickel was annealed to attain a constant line sharpness has been plotted in arithmetical coordinates against the corresponding time in minutes as logarithmic abscissae. Thus plotted, the points follow a straight line within the experimental error. Van Liempt (18) assumes that recrystallization consists of a place change of the fraction of atoms in the distorted metal lattice whose energy corresponds to a certain minimum vibration amplitude, and that the

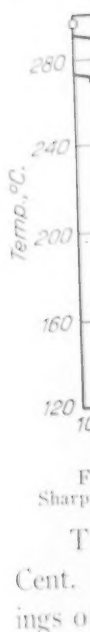
"Springzeit" (jumping time) of a diffusing atom is $B = \frac{1}{4\nu}$, where ν is the characteristic vibration frequency according to Einstein's specific heat theory. By analogy with kinetic gas theory, he then shows that the time of place change in all directions for the majority of lattice points is

$$t = \frac{1}{6} \cdot \frac{3}{2\nu} \cdot e^{\frac{3b^2T_s}{T_R} - \frac{K\beta}{T_R}}$$

where K and b are constants, T_R and T_s are, respectively, recrystallization and melting temperatures in absolute Centigrade units and β is a measure of the degree of deformation. From this, for a given specimen of cold-worked metal,

$$T_R = \frac{\text{constant}}{\log_{10} t - \log_{10} \frac{1}{4\nu}}$$

For the case of recovery from line broadness, van Liempt's assumption that the vibration time for place change is equal to one-fourth the period of the atom may be modified, since the atoms are displaced from their normal positions by rather small amounts in relation to the interatomic distance. However, the data of Table VI show constant values within 1.5 per cent of the mean if the values for the $\frac{1}{2}$ - and 1-hour anneals are omitted. Since it was noted that the latter recovery curves tended to flatten with increased annealing temperatures, it is entirely possible that the extrapolations are in error for the short-time anneals.



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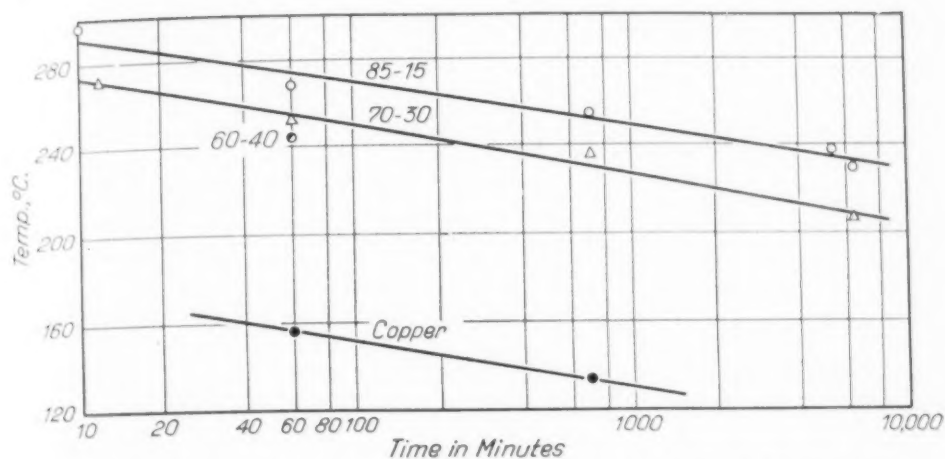


Fig. 23—Time and Temperature of Annealing for Recovery of Constant Line Sharpness in Copper and Copper-Zinc Alloys.

The series of annealing experiments conducted at 512 degrees Cent. (955 degrees Fahr.) show (Fig. 9) that contrary to the findings of Burgers and van Arkel (8) on tungsten, the line sharpening

Table VI*

t (sec.)	T_R (Deg. C. abs.)	$T_R (\log_{10} t + 13.50)$
1,800	855	14,300
3,600	845	14,500
18,000	838	14,900
93,600	822	15,300
151,200	805	15,000
345,600	799	15,200

*The value for $-\log_{10} \frac{1}{4\nu}$ is derived from Lindemann's equation, which yields, $\nu_{N1} = 8.1 \times 10^{12}$.

of nickel at low annealing temperatures consists not only of the rapid initial drop noted by these authors, but also a long induction period followed by a gradual sharpening corresponding to a diffusion process. This indicates that at the chosen annealing temperature the fraction of atoms whose energy content is sufficient for diffusion

$$= K_1 e^{\frac{K_2\beta}{T_R} - \frac{b^2T_s}{T_R}}$$

is small for heating times less than 26 hours.

ALLOY SYSTEMS

Copper-Zinc—The effect of annealing time on temperature of line sharpening was studied in the 85-15 and the 70-30 brass. The results are plotted as for pure nickel in Fig. 23, together with the data

obtained on 60-40 brass and copper. The experimentally derived constants for these curves corresponding to those of the equation for nickel are summarized in Table VII.

Table VII

Material	Constant	$-\log_{10} \frac{1}{4n\nu}$
Copper	6,800	15.7
85-15 brass	13,100	14.5
70-30 brass	10,061	15.3

The fact that the vibration constant is somewhat larger than predicted by use of van Liempt's assumption is in accord with the nature of the recovery from line broadness. In this process it is thought that recovery consists of return to normal positions rather than a place change as conceived by van Liempt.

Relation of Line Broadness to Season Cracking in 70-30 Brass—

It is well known that copper-zinc alloys in the cold-worked state are subject to failure by the formation of spontaneous cracks. There has been much speculation concerning the cause of this type of failure. Many investigators have shown that annealing at temperatures under the softening point is sufficient to remove the cause of such failures. It has been the general consensus of opinion among metallurgists that internal stresses of some sort were the cause of the season cracking phenomena, but the exact nature of these stresses has never been satisfactorily explained.

Moore and Beckinsale (20) in 1920 made a thorough investigation of the times at various annealing temperatures necessary to prevent accelerated cracking in mercurous nitrate solution. Their results showed that the temperature for the attainment of this immunity to mercury salts was definitely decreased as the annealing time is increased. They did not, however, attempt to formulate a quantitative time-temperature relationship due perhaps to the experimental spread in their results. However, if their data are plotted with the time in logarithmic and the temperature in arithmetical units, and a line drawn through the points thus obtained, the solid curve of Fig. 24 results. The broken line curve is a reproduction of the line sharpening curve for 70-30 brass in Fig. 23 plotted to the same scale as the mercury tests. It is also interesting to note the close agreement obtained on the 1-hour test of W. B. Price (21) to the 1-hour line sharpening temperature of the present investigations. While the slopes of the two lines do not agree particularly, the temperature

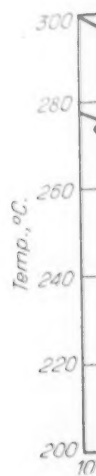


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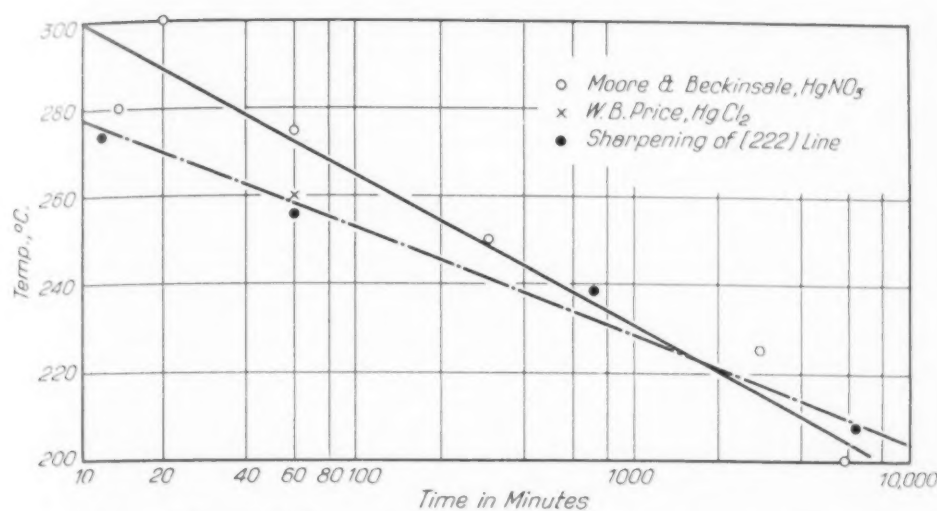


Fig. 24—Removal of Internal Stress in 70-30 Brass as Shown by Sharpening of Hull-Debye Lines and by Accelerated Cracking Tests in Solutions of Mercury Salts.

range and trend is the same for both sets of data. A much better agreement in slope would have been obtained had the 20-minute and 100-hour points been omitted from Moore and Beckinsale's data.

The close agreement of the line broadness data with the results of Moore and Beckinsale indicates that the internal stresses which cause season cracking are somehow associated with the line broadening. This in itself does not clarify the mechanism by which brass fails spontaneously on aging after cold work, but the phenomenology of season cracking can be satisfactorily explained in terms of the hypothesis of the present thesis with the knowledge of these facts.

In the periodic bending or crumpling of the lattice segments which give rise to the broadening of X-ray lines, the distances between various atom pairs, or even in the whole set comprising the block, are changed. This strain, of course, in mechanical terminology is accompanied by an equivalent stress, provided it is elastic in nature. Calculations by Caglioti and Sachs (10) have shown that only a few per cent of the energy stored in the lattice can be ascribed to elastic stress. However, this distortion may also affect the atoms in such a way that the bondings of molecular type are increased, thus storing added energy in unstable molecular configurations. The energy content of the various atoms in a block is distributed in a statistical manner, with the most highly distorted atoms probably in the boundary layers. Investigation¹ of electrical conductivities has shown that

¹Tammann and Dreyer, *Ann. der Phys.*, Vol. 16, 1933, p. 657, noted a 2-stage recovery of electrical conductivity in copper-zinc alloys with inflection points at 80 and 220 degrees Cent. (430 degrees Fahr.) for the 28 per cent zinc alloy.

changes in the degree of binding of the valence electrons begin to take place at relatively low temperatures. This is particularly evident in the copper-zinc alloys which show a recovery effect which may apparently be ascribed to the zinc atom at temperatures in the neighborhood of 100 degrees Cent. for 1-hour anneals. In view of the results obtained by Goetz and Focke (22) on bismuth alloys, this may indicate that the zinc atoms are concentrated on certain atomic planes designated as π planes. If such a structure were assumed for brass, such planes would be the most favorable for slip; thus, the boundary planes of slip lamellae are probably higher in zinc than the body of the slip lamellae.

On heating, the energy distribution of a block tends naturally toward a level. However, due to the restrictions imposed by the boundaries, the readjustments evidenced by freeing of valence electrons (increased electrical conductivity) lead to an actual increase in energy at the boundary planes.

The assumption of this state is a corollary to the fact that the change of line broadness is very small, indicating that the total amount of distortion is practically unchanged. This increase in energy of the boundary atoms is used as an increase in the molecular bondings of these atoms, thus building up local stress concentrations at grain boundaries which exceed the tensile strength of the alloy.

On annealing at higher temperatures, the second stage in recovery, made possible by the loosening of interatomic bindings, occurs. This is the actual diffusion of displaced atoms to normal position as evidenced by the sharpening of lines. When this sharpening reaches a certain stage, the boundary atom deformations begin to disappear. *It is the inception of the latter state of the recovery process which is of importance in the elimination of "season cracking" tendencies in brass.* As was previously shown, the temperature difference between the boundary atom recovery and the general lattice recovery is small for this metal. This indicates the reason for the similarity of temperature for the definite decrease in chemical reactivity of the boundary atoms as shown by the mercury salt tests, and the recovery from lattice distortion as shown by the line broadness data.

Relation to Creep Properties

In the preceding discussion it was pointed out that the line sharpness recovery temperature of the brasses possessed a maximum

at an intermediate composition in the alpha range. This is brought out more clearly in Fig. 25, which shows the results for the 1-hour anneals on copper and the brasses plotted in relation to the zinc content. It is seen that for the alloys studied the recovery temperature is at a maximum for 15 per cent zinc. This is in agreement, not only with the electrical conductivity results of Tammann and Dreyer, who

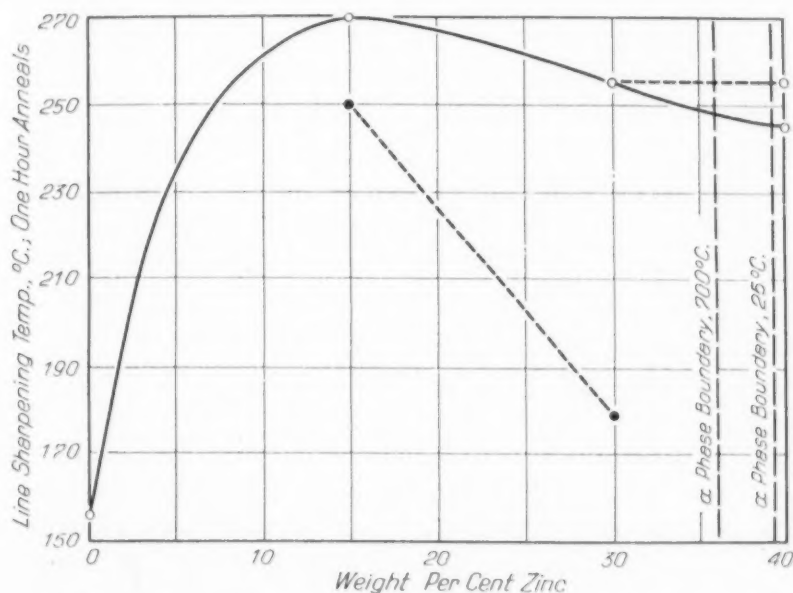


Fig. 25—Copper-Zinc Alloys. Relationship of Zinc Content. Line Sharpening and Creep.

found the recovery range highest in the 10 per cent zinc alloy, but also with some experiments on hot hardness performed by Meneghini (23) and by Doerinckle and Trockele (24). The former found that the alloy containing 20 per cent zinc required the highest temperature for penetration of a steel cone under constant pressure, the latter that for 50 per cent compression, minimum compressibility occurred at approximately 15 per cent zinc. These results are also confirmed by the work of Clark and White (25) on the creep characteristics of the alloys used in this investigation. Their data for creep at 315 degrees Cent. (600 degrees Fahr.) are also reproduced in Fig. 25 for the 85-15 and the 70-30 compositions. The results for the 60-40 composition were not obtained at this temperature, but from the low value obtained at 205 degrees Cent. (400 degrees Fahr.), it is believed that the stress for creep of the same magnitude would be only a few pounds.

The results obtained by Clark and White would not have been

so marked in their difference in stress values for the 0.01 per cent strain per 1000 hours at 315 degrees Cent. (600 degrees Fahr.), had the 85-15 and the 70-30 materials been in similar initial condition. The 70-30 material had been cold drawn previous to testing. Clark and White² have found in the case of a carbon steel that stretching at room temperature previous to the creep test has the effect of marked reduction of the limiting creep stresses at temperature from 800 to 1000 degrees Fahr. (425 to 540 degrees Cent.). The cold-drawing increases the tendency toward recovery at the test temperature, and thus upsets the equilibrium between the rates of isothermal strain hardening and recovery which normally obtains in the creep test. However, the difference in creep stress between the 85-15 and 70-30 would seem to be too large to be ascribed entirely to this cause, particularly in view of the evidence on hot-hardness and X-ray line sharpening. It may accordingly be concluded that there is some factor in the addition of zinc to copper which causes the maximum stability at high temperatures to occur at a composition somewhere in the neighborhood of 15 per cent zinc.

Copper-Nickel Alloys—The relation of temperature of line sharpening at constant annealing time to the relative creep properties of a series of alloys is substantiated by the relation between these factors for the Adnic and Monel metal compositions. The creep properties at various temperatures for these alloys have been obtained by Clark and White.³ They are summarized, together with the line sharpening temperature of the respective alloys, in Table VIII.

Table VIII
Creep Properties

Stress for Creep of 0.01 Per Cent per 1000 Hrs. at T. Deg. C.		Inflection Points of Line Sharpening Curves	
T Deg. C.	Pounds per Square Inch	T Deg. C.	
315	26,000		
Monel 427	19,000	590	
538	1,600		
315	13,800		
Adnic 427	4,500	450	
538	630	570	

It is apparent that both the high temperature stability and the line sharpening temperatures are a maximum for the Monel metal composition, which incidentally confirms the findings on the brasses that the maximum temperature stability is found near the middle of the continuous series of solid solutions in terms of atomic percent-

²Private communication.

³Clark and White, *Transactions*, American Society of Mechanical Engineers, Vol. 53, 1931, FSP-53-15, p. 183-191.

ages. It is believed that while the actual values for creep stresses may be affected by the impurities other than copper and nickel in these alloys, the trend of the values is controlled by the main components, particularly in solid solution alloys of this type.

Pearlitic Alloyed Steels of Varying Manganese Content—It was shown in the experimental part that the series of steels ranging from one to three per cent manganese exhibited a unique phenomenon on annealing the cold-worked specimens in the temperature range 400-500 degrees Cent. (750-930 degrees Fahr.). The alloys in general showed an initial recovery from line broadening which was, however, interrupted by a line-broadening lattice distortion from another cause on annealing at slightly higher temperatures. This secondary lattice distortion was accompanied or paralleled by an increase in Rockwell hardness which indicates that the noted X-ray phenomena are probably due to a precipitation hardening effect.

Before proceeding to a discussion of the possible causes and implications of the observed precipitation effect, the effect of the addition of molybdenum and vanadium to the ternary iron-manganese carbon alloy may be reviewed. In Fig. 26 are reproduced the X-ray recovery curves for steels MM6, MM2 and QV, all containing approximately 2 per cent manganese. MM2 and QV contain in addition 0.21 and 0.32 per cent molybdenum, respectively, and QV also has a vanadium content of 0.20 per cent. In comparing these curves, it is apparent that 1, the additional alloys have the effect of: Reducing the slope of the initial recovery from line broadening. 2, of Displacing the secondary broadening phenomena to higher temperature regions.

The effect "1" is quantitatively expressed in Table IX, which indicates the slopes $\Delta B/\Delta T$ where B is line broadness and T is tem-

Table IX

Steel	Effect of Alloys on		$\frac{\Delta B}{\Delta T}$		$\Delta B/\Delta T$
			T_1	T_2	
MM6	B_1	B_2	400	425	0.188
MM2	11.6	6.9	400	450	0.0881
QV	12.7	8.3	350	475	0.0368
	11.5	6.9			

perature. Since the molybdenum content is increased 50 per cent in addition to the vanadium content, it is impossible to evaluate the effect of vanadium in decreasing the slope of the QV alloy.

In the analysis of this decrease in slope, however, the effect of the secondary broadening phenomenon should be particularly considered. The temperature range of the initial recovery from line broadness is conditioned by two factors in these alloys. First, the inherent characteristics of the recovery from cold-working, which

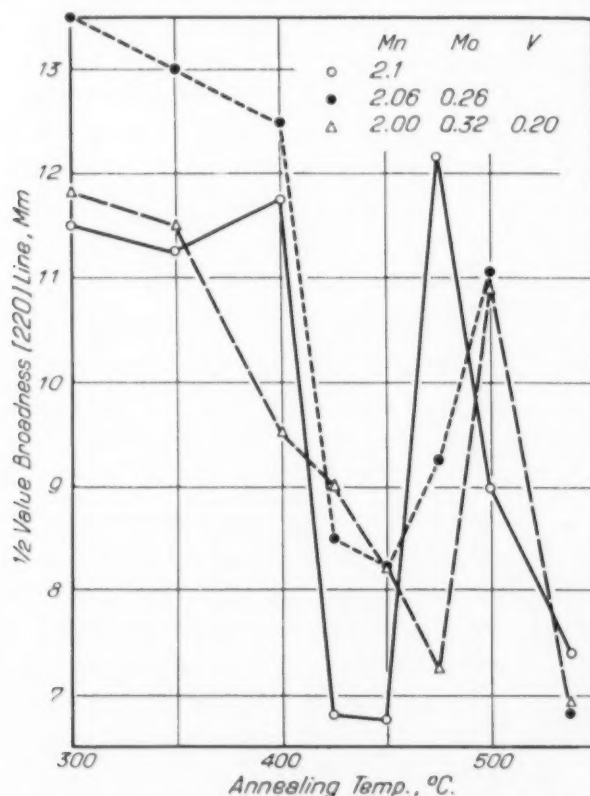


Fig. 26—X-Ray Recovery Curves for Steels. One-Half Value Broadness (220) Line MM.

has been shown to differ widely in the pure metals; and second, the temperature at which the precipitation effect becomes noticeable in the line broadening. These overlapping effects are shown in the hypothetical curves reproduced in Fig. 27.

In Fig. 27, Curve 1 represents the normal recovery trend of the solid solution alloy, Curve 2 the line-broadening at various annealing temperatures due to precipitation effects, and Curve 3 is the summation curve of 1 and 2. Curve 3 is the experimentally observed effect. From this picture it is seen that the position of the minimum point "a" is dependent on the temperature of the maximum point "b" and upon the slope of the curves 1 and 2 near the point of intersection. These variations are apparent in the changed appearance of the man-

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ganese-molybdenum series as the manganese content is increased. In the 1.1 per cent manganese alloy, practically no precipitation effect is present, and a normal curve of recovery from line-broadening caused by cold work results. The 1.25 per cent manganese steel indicates that the secondary broadening is approaching its maximum before

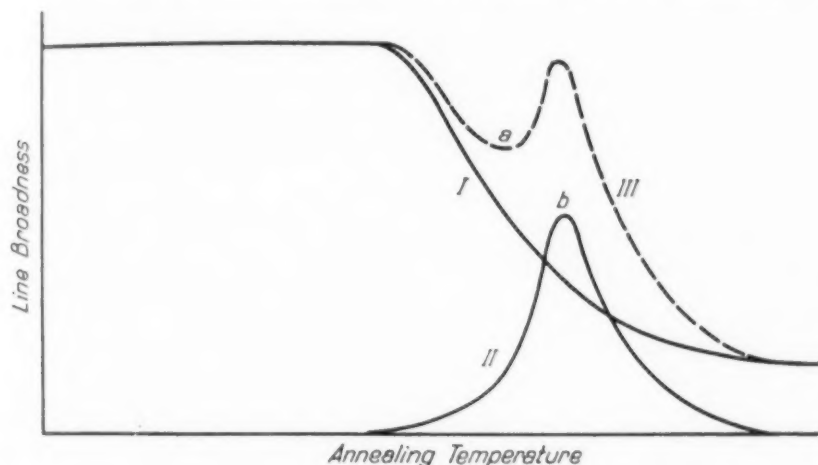


Fig. 27—Overlapping Effects as Shown by Hypothetical Curves.

recovery from line broadness due to cold work has reached a rapid rate. This means that either Curve 1 is displaced toward higher temperatures, or that Curve 2 is displaced toward lower temperatures, or both. In the light of other experimental evidence such as the small magnitude of the hardness change and the temperature at which it occurs, one is inclined to the former hypothesis. In the next alloy of the series a distinct separation of the two effects is first evident. The MM9 alloy containing 1.75 per cent manganese shows a distinct tendency for the precipitation effect to be displaced toward higher temperature regions. This temperature effect is not proportional to manganese content, since the temperature of the maximum drops to 450 degrees Cent. (840 degrees Fahr.) in the 2.0 per cent manganese steel; although, as the hardness measurements show, the magnitude of the precipitation effect increases with the manganese content. For instance, in the MM8 (1.25 per cent manganese) steel the precipitation hardening amounts to only a 0.7 point Rockwell "B" increase, while the MM2 (2.06 per cent manganese) has a total increase of 3.5 points Rockwell "B."

These considerations lead to two possible explanations of the precipitation effect:

1. The iron-manganese solid solution, due to heterogeneities, is precipitating the epsilon phase, or

2. Precipitation of complex carbides of manganese occurs on annealing the cold-worked alloys.

In support of the first hypothesis, the recent publication of Walters (26) and co-workers on the binary iron-manganese alloys indicates that marked heterogeneity of composition within the individual crystals may occur in slowly cooled alloys and that the tendency toward the formation of epsilon is increased by cold-working. This would make the precipitation of epsilon particularly favorable under the present experimental conditions.

In support of the second hypothesis, the work of W. A. Wood (27) on broadening of lines on annealing tungsten magnet steels caused him to conclude that the broadening in those steels was due to carbide precipitation. Cold work is known also to cause an increased tendency toward carbide precipitation, and the effect of molybdenum and vanadium in displacing the secondary broadening effect toward higher temperature ranges might be taken as an indication that the carbides are the precipitated phase, inasmuch as these alloying elements are known to be remarkable carbide stabilizers. This evidence is not conclusive, however, since the unknown effect of molybdenum and vanadium on the stability of the iron-manganese solid solution is also involved.

A third possibility, that the observed effect is due to a combination of the hypotheses offered above, seems entirely reasonable; the latter hypothesis is perhaps the best compromise obtainable in the lack of entirely conclusive evidence.

These alloys have been subjected to extensive high temperature creep studies by A. E. White and C. L. Clark, of this laboratory. In view of the correlation obtained of creep results with the X-ray studies, in the case of the copper-zinc alloys, it is more or less to be expected that some light may be thrown on the relative creep properties of the manganese steels by the results of the present investigation. The creep test data on the steels of the MM series obtained at 900 degrees Fahr. (480 degrees Cent.) are summarized in Table X.

These data are graphically presented in Fig. 28, along with the X-ray line broadness results. It will be noted that the first maximum in the creep stress curve at 900 degrees Fahr. (480 degrees Cent.) occurs at the same composition as the maximum temperature of initial sharpening, the second maximum coincides with composition of

Table X
Stresses for Creep of 0.01 Per Cent per 1000 Hours at 900 Degrees Fahr.
(480 Degrees Cent.) in Selected Manganese-Molybdenum Steels

Designation	Per Cent Manganese	Stresses Pounds per Square Inch
MM10	1.10	10,200
MM8	1.25	21,000
MM1	1.46	10,200
MM9	1.75	16,000
MM2	2.06	9,600
MM4	2.99	7,000

alloy which has the highest temperature of the inception of line broadening effect.

This indicates that the creep properties of the manganese-molybdenum steels are due partially to the properties of the normal solid solution and pearlite mixture and partially to the precipitation effect. If the precipitation is excluded from consideration, the 1.25

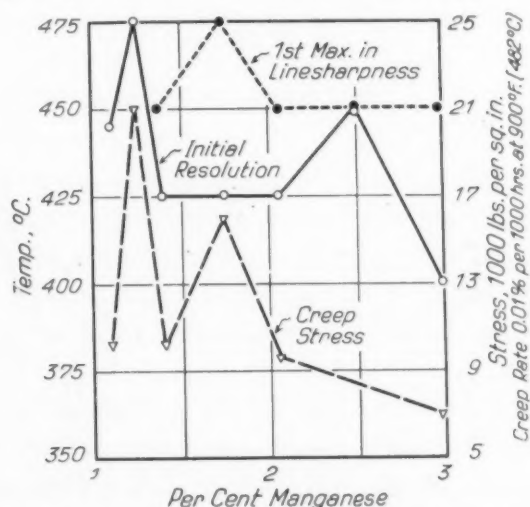


Fig. 28—Line-Sharpening Temperatures and Creep Stress at 90 Degrees Fahr. of Manganese-Molybdenum Steels on Annealing as a Function of the Manganese Content.

per cent manganese composition seems to be the alloy of critical composition from the standpoint of temperature stability of strength; this alloy may be compared to the 85-15 brass of the copper-zinc system. In the same way that the greater temperature stability of that alloy was shown by its slightly higher recovery temperature from X-ray line broadening, so the 1.25 per cent manganese steel shows that its solid solution is of the optimum composition by the extension of its recovery range to such high temperatures that the precipitation effect sets in before any sharpening is observed.

The 1.75 per cent manganese steel exhibits the effect of a critical concentration of precipitating substances for attainment of long time strength at 900 degrees Fahr. (480 degrees Cent.).

The high temperature properties of the QV steel are of interest since this steel has almost identical creep properties at 900 degrees Fahr. (480 degrees Cent.) to the MM8 composition. The logarithmic

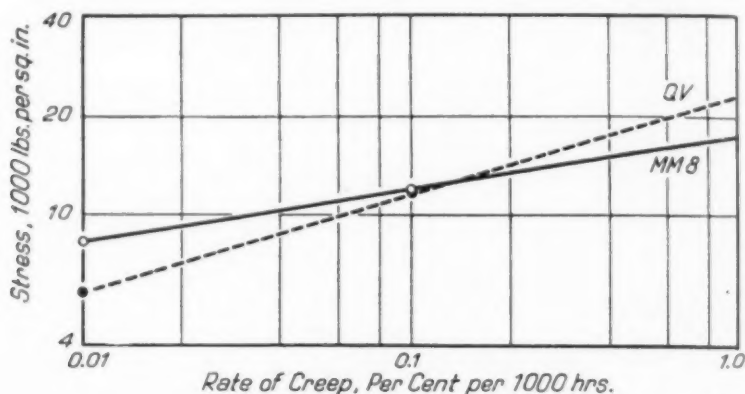


Fig. 29—Creep-Stress Curves for MM8 and QV Steel at 1000 Degrees Fahr.

creep stress curves for these two compositions are reproduced in Fig. 29. It is apparent that the MM8 is somewhat superior at 1000 degrees Fahr. (540 Cent.) for the low rate of creep (0.01 per cent per 1000 hours) but that the QV has a greater slope and becomes equal in strength to MM8 at a creep rate of 0.1 per cent per 1000 hours.

The line broadness recovery curve of the QV steel indicated initial recovery at a much lower temperature than the MM8 but showed a very broad temperature range of recovery, and a precipitation effect displaced toward higher temperatures than most of the steels investigated. A low rate of line sharpening may thus indicate a greater tendency toward strain hardening at elevated temperatures.

SUMMARY

1. The broadness of Hull-Debye-Scherrer lines of selected cold-worked metals and alloys has been investigated as a function of temperature and time of annealing. The effect of deformation temperature on the broadness of lines of aluminum and copper was also investigated. The recovery from line broadness on annealing can be treated as a diffusion process. At low annealing temperatures the recovery is characterized by a long induction period followed by a

gradual increase of line sharpness. Results on nickel, copper and 85-15 and 70-30 brass indicate the following relation between time and temperature of annealing for attainment of constant line sharpness:

$$T_R = \frac{\text{constant}}{\frac{1}{\log_{10} t - \log_{10} t_0 - 4n^2}} .$$

2. The results have been correlated with electrical conductivity, magnetic susceptibility and hardness data. It is shown that the lattice distortion responsible for most line broadening is of a different nature than that involved in the hardness and electrical changes. The electrical conductivity changes on cold-working are due to interatomic binding of "free" electrons in the body of the lattice due to distortion of electron shells, while hardness and magnetic changes are due to similar forces acting on grain and slip lamella boundary atoms, according to the writers' hypothesis.

3. It was shown that line sharpening of 70-30 brass closely parallels existing data on relief anneals for elimination of season cracking tendencies. The hypothesis is advanced that season cracking is due to redistribution of interatomic forces leading to abnormal cohesion forces at grain boundaries.

4. Correlation with existing creep data shows that maximum creep properties coincide with maximum line sharpening temperatures. Similar parallelism is indicated by experiments on Admic and Monel metal. The poor temperature stability of 60-40 brass is shown to be due to the fact that the recovery temperature of the beta phase is distinctly lower than that of the alpha. The use of the X-ray method is thus shown to be applicable to the analysis of recrystallization phenomena in complex alloys in which the microscopic evidence is confusing.

5. Recovery of a series of cold-worked pearlitic steels of varying manganese content from line broadness was investigated. It was found that there is superposed on the normal recovery curve a re-broadening due to a precipitation hardening effect. Correlation with creep data of White and Clark shows that first composition of optimum strength at 900 degrees Fahr. (480 degrees Cent.) (1.25 per cent manganese) coincides with maximum temperature of line sharpening, and that a second composition (1.75 per cent manganese) which also has exceptional creep characteristics corresponds to maximum temperature for inception of precipitation effect.

6. Effect of additions of molybdenum and vanadium to the manganese steels is to reduce rate of line sharpening with respect to temperature, indicating that diffusion of atoms of cold-worked iron-manganese solid solution is inhibited by these alloys.

7. The correlations with creep data indicate that the line sharpening temperatures of a given series of alloys may be an indication of their relative creep characteristics.

ACKNOWLEDGMENTS

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They are especially grateful to Prof. A. E. White and Dr. C. L. Clark for permission to use certain unpublished creep data, and to the latter for many helpful discussions.

The Departments of Chemical Engineering and Engineering Research of the University of Michigan very generously provided Mr. Wilson with research assistantships, under the terms of which the experimental work was carried out.

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EFFECT OF THERMAL STRESSES ON AUSTENITE

BY H. P. NIELSEN AND R. L. DOWDELL

Abstract

Stresses apparently play an important part in the transformation of austenite to martensite. In the following study, stress distribution has been simplified by the use of steel balls, while thermal stresses are made negligible through the use of very slow cooling rates. It has been found possible to retain austenite at 100 degrees Cent. in carburized ingot iron as well as in a white cast iron.

INTRODUCTION

IN spite of the fact that an enormous amount of work has been done on the decomposition of austenite and the formation of martensite, there are apparently a number of questions, some seemingly simple, which have not yet been satisfactorily answered. Possibly, some of these problems will never be solved to the satisfaction of everyone. Much of the controversy has been caused by a mere lack of agreement on definitions. As an example, some investigators define martensite as those needles which remain a blue white on etching, and yet many authors draw conclusions concerning this constituent from photomicrographs in which not one white needle can be observed.

It is generally agreed that stresses play a rather important part in the heat treating of steels, particularly when such stresses are of a magnitude sufficient to cause fracture. There are, however, other and more complicated effects of stress which undoubtedly have an important bearing on the various allotropic and other changes in steel and the complete determination of these effects is a problem not so easily solved.

This paper is based upon a thesis presented to the University of Minnesota in partial fulfillment for the degree of Doctor of Philosophy. 1933.

Of the authors, H. P. Nielsen is research assistant in the Department of Metallography, University of Minnesota and R. L. Dowdell is Professor of Metallography, University of Minnesota, Minneapolis, Minn. Manuscript received April 6, 1934.

From a review of the literature it will be apparent that the rather definite facts on austenite and its decomposition products are:

1. Austenite is an interstitial solid solution of gamma iron and carbon having a face-centered cubic lattice. A hypothetical gamma iron of high purity has a lattice parameter of about 3.60 Å.U. at room temperature and a density of about 8.0.

2. Ferrite is alpha iron in which small amounts of impurities such as C and N may be contained in interstitial solid solution while elements such as Ni, Cr, Mn would be substitutional. Its lattice is body-centered cubic with a lattice constant of about 2.86 Å.U. and a density of about 7.86.

3. Cementite is iron carbide, Fe_3C . It crystallizes in the orthorhombic system and has parameter values of about $a_1 = 4.51$ Å.U., $b_2 = 5.08$ Å.U. and $c_3 = 6.73$ Å.U. Its density is about 7.67.

4. Martensite is the acicular constituent the formation of which is the cause of the hardening phenomena during the usual quenching of steel. Unless tempered, it remains white and in relief when etched with the usual acid reagents. A body-centered tetragonal form of martensite exists, the parameter values being a linear function of the carbon content. The density of martensite is less than that of any of the other constituents found in steel.

5. Troostite is an aggregate of submicroscopic particles of cementite and ferrite which appears black almost immediately upon etching with the usual acid reagents.

6. All quenched steels contain some austenite.

7. The amount of retained austenite increases with

(a) increase in its carbon content

(b) increase in alloying elements.

8. More austenite is usually retained by oil quenching than by more drastic quenching in water or a 5 per cent solution of sodium hydroxide in water.

9. Martensite decomposes slowly at about 100 degrees Cent.

10. Austenite decomposes to martensite (tetragonal lattice) in liquid air, although the reaction does not go to completion.

11. Austenite decomposes on tempering, usually to troostite, but its decomposition temperature is from 100 to 150 degrees Cent. higher than the decomposition temperature of martensite.

12. In a steel of high carbon content, austenite may be retained by quenching to a temperature just above that at which mar-

tensite begins to form (Ar'') and will be stable for a considerable time if maintained at this temperature. On further cooling to room temperature, some martensite is formed, but the reaction does not go to completion.

13. The stability of austenite is decreased by cold deformation and some recrystallization to alpha takes place.

14. Martensite is formed along the octahedral planes of the parent austenite.

15. The surface of a quenched piece of steel is usually in compression and the inside in tension. Cracking is caused primarily by tensional stress.

16. Stresses play some part in the retention of austenite and the formation of martensite.

The authors are interested primarily in the effects of stress on austenitic decomposition and have made experiments reported here which should throw more light on this phase of the subject.

BRIEF RÉSUMÉ OF RECENT LITERATURE

The literature on austenite decomposition up to the year 1927 has already been carefully reviewed by Dr. O. E. Harder and one of the authors.¹ In their paper the effect of stress, liquid air immersion and tempering on austenite decomposition were studied for two carbon steels and eight alloy steels, and the following results obtained:

More austenite was retained when the steels were quenched from a high temperature than from the normal quenching temperature. In the smaller specimens ($\frac{3}{8}$ -inch diameter) more martensite was found at the edge of the piece than on the inside.

On immersion in liquid oxygen, the amount of decomposition varied greatly for the different steels, but in the plain carbon steel (1.35 per cent carbon) it went practically to completion. The needles produced were found to be similar to those formed by quenching. Most of the decomposition was found to take place during the first twelve hours of immersion, and very little after that.

On tempering, the martensite needles in the carbon steels were found to decompose at 100 degrees Cent., but were more stable in the alloy steels. The decomposition temperature of austenite was found in every case to be higher than the decomposition tempera-

¹R. L. Dowdell and O. E. Harder, TRANSACTIONS, American Society for Steel Treating, Vol. 11, 1927, p. 217-32; 391-8; 583-606; 781-90; 959-74; Vol. 12, 1928, p. 51-68.

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ture of martensite—150 to 200 degrees Cent. (300-390 degrees Fahr.) for the plain carbon steel.

In all cases, cold deformation made the austenite less stable when subjected to tempering or liquid air immersion. In the case of a low tungsten tool steel martensite was produced from austenite by the mere application of tensile stress.

Robertson² studied the microstructure of carbon steels quenched in molten metal at different temperatures from 220 to 600 degrees Cent. (430-1110 degrees Fahr.). He concluded that the crystallographic form of the product of the decomposition of austenite depended on whether the decomposition was initiated by the allotropic change or by the formation of cementite. He stated that the Ar' was initiated by cementite while the Ar'' was initiated by the allotropic change.

Lewis³ found that specimens of 0.8 per cent carbon steel quenched in salt at 232 degrees Cent. on withdrawal from the salt were soft and nonmagnetic, but on further cooling in air became hard and brittle.

Bain and Waring⁴ measured the length changes for an oil hardening die steel and a stainless steel for various quenching rates and plotted curves showing length change as a function of time. They found that the higher the quenching temperature the greater was the initial contraction. They also found that an oil quench caused a greater initial contraction than a water quench. They found the final length was greater with water quenching than with oil quenching and concluded that more austenite was retained by quenching in oil due to the smaller tensile stresses.

They also thoroughly insulated a steel bar containing a lot of residual austenite, sealed it into a 4-inch pipe, immersed it in liquid air for several hours and then allowed it to come gradually to room temperature again. The hardness, length change and magnetic properties were then measured, whereupon, the bar was then quickly cooled by direct immersion to liquid air temperature and the measurements again taken. The first refrigeration caused a considerable change while the second caused substantially none. They concluded that whatever change was brought about by very low temperatures was due to a steep thermal gradient and the accompanying stresses.

²Robertson, *Journal, Iron and Steel Institute*, Vol. 119, No. 1, 1929, p. 391-419.

³Lewis, *Journal, Iron and Steel Institute*, Vol. 119, No. 1, 1929, p. 427-437.

⁴Bain and Waring, *TRANSACTIONS, American Society for Steel Treating*, Vol. 15, 1929, p. 69-90.

On quenching a series of specimens of carbon steel containing 0.9 to 1.7 per cent carbon, Honda and Osawa⁵ found by the intensity of medial lines from X-ray photograms that more austenite was retained on the outside of the specimen than on the inside.

Scheil⁶ concluded that the formation of martensite from austenite was due to thrust strains.

Maurer and Schroeter,⁷ in a plain carbon steel of eutectoid composition, found that more austenite was retained by water quenching than by oil quenching. The reverse was found to be true for a number of alloy steels. The decomposition temperature of martensite was given at 100 degrees Cent. while that of austenite was 200 to 250 degrees Cent. (390-480 degrees Fahr.).

In an investigation on a magnet steel containing 5.7 per cent tungsten, Van Vleet and Upthegrove⁸ found that more austenite was retained by quenching from the higher temperatures. Specimens quenched in oil from above 870 degrees Cent. (1600 degree Fahr.) had a greater density than those quenched either in water or in a 5 per cent solution of sodium hydroxide in water. Austenite retained in the bars quenched from the higher temperatures showed a greater degree of stability upon liquid air immersion than did those quenched from the lower temperatures.

Sauveur⁹ was able to produce needles resembling martensite on the surface of a piece of electrolytic iron heated to 1000 degrees Cent. (1830 degrees Fahr.) in vacuum and quenched in ice cold mercury. He pointed out the similarity between the Widmanstätten structure produced by slow cooling and the martensitic structure produced by rapid cooling. He thought it likely that ordinary martensite needles contained finely divided cementite and so suggested that the needles be called troostite and that martensite be regarded as an aggregate of troostite and austenite.

Davenport and Bain¹⁰ made some quenching experiments on three carbon steels, three low manganese steels, a razor blade steel and a cutlery stainless steel. The specimens were quenched from

⁵Honda and Osawa, *Science Reports*, Tohoku Imperial University, Vol. 18, 1929, p. 47-58.

⁶Scheil, *Zeitschrift für Anorganische und Allgemeine Chemie*, Vol. 183, 1929, p. 98-120.

⁷Maurer and Schroeter, *Stahl und Eisen*, Vol. 49, 1929, p. 929-40.

⁸Van Vleet and Upthegrove, *TRANSACTIONS*, American Society for Steel Treating, Vol. 18, 1930, p. 729-759.

⁹Sauveur, *TRANSACTIONS*, American Society for Steel Treating, Vol. 17, 1930, p. 199-218.

¹⁰Davenport and Bain, *American Institute Mining and Metallurgical Engineers, Technical Publication No. 348*, 1930.

above the critical to different temperatures below the critical and held at the quenching temperature for varying lengths of time. No austenite was found in the products of transformation above 180 degrees Cent. The amount of retained austenite increased rapidly with increase in carbon content, the amount retained being 20 to 25 per cent in the higher carbon steels (1.13 and 1.17 per cent carbon) and only a trace in the carbon steel with 0.54 per cent carbon. They found that "austenite may be definitely retained permanently in steels in which the ultimate transformation product is not martensite-troostite structures."

Tamaru and Sekito¹¹ devised a method for determining the amount of retained austenite by X-ray analysis, using the intensity of the austenite line in an austenitic manganese steel as a standard.

Robertson¹² stated that "the formation of martensite is not caused by stress. It is primarily a constitutional phenomena." When a high carbon steel was quenched from 1100 degrees Cent. in molten metal at 200 degrees Cent. (390 degrees Fahr.) all of the austenite was retained in spite of the necessarily high thermal stresses which existed. On the other hand, martensite was formed under further slow cooling to room temperature under conditions where the stress must have been very slight. He said, however, that thermal stresses might facilitate the retention of austenite. He claimed it was mainly micro-stresses (previously mentioned by Scott¹³) that caused austenite to be retained in high carbon steels, the martensite simply forming to offset the normal shrinkage of the austenite during cooling. He stated that the product of the decomposition of austenite must be similar to that of the decomposition of martensite, since austenite retained by quenching decomposes during tempering in the same way as austenite quenched into a medium held at tempering temperature.

Greene¹⁴ used the method of Heyn to calculate the axial stresses existing in quenched hollow cylinders of carbon tool steel and derived a method for calculation of the tangential and radial stresses. On quenching by ordinary immersion the inside surface was found to be in tension while the outside surface was in compression. On

¹¹Tamaru and Sekito, *Science Reports*, Tohoku Imperial University, Vol. 20, 1931, p. 377-94.

¹²Robertson, *Carnegie Scholarship Memoirs*, Vol. 20, 1931, p. 1-64.

¹³Scott, *Scientific Paper*, U. S. Bureau of Standards, No. 513, 1925.

¹⁴Greene, *TRANSACTIONS*, American Society for Steel Treating, Vol. 18, 1930, p. 369-403.

hardening by flushing at the hole, however, the center of the hole was in compression.

Wever and Möller,¹⁵ Regler have developed methods for estimating internal stresses by means of the X-ray. Recently, Berthold suggested a new method utilizing the X-ray back reflection principle which he claims to have an accuracy of ± 5 kilograms per square millimeter.

Steinberg, and Steinberg and Zuzin,¹⁶ studied the decomposition of retained austenite in steels containing from 0.62 to 1.60 per cent carbon and concluded from magnetic analysis that the amount of residual austenite on quenching increased with increase of carbon. They found that a rise of quenching temperature increased the austenite content up to 950 to 1000 degrees Cent., and then decreased the content. The slower quenches produced more austenite up to 1.6 per cent carbon. Above 1.60 per cent carbon higher speed produced more austenite.

Wever and Engel¹⁷ investigated the effects of different cooling velocities on A_3 , A_2 and A_1 . For carbon contents above 0.3 per cent these appeared as one effect. At 0.037 per cent carbon a new point M appeared at about 440 degrees Cent. for cooling velocities above 5300 degrees Cent. per second. This point was lowered with increasing carbon and increasingly became weaker. In a 1.8 per cent carbon steel M is but faint at 120 degrees Cent. They offered evidence to show that M corresponded to the appearance of a new tetragonal crystal. They found that M was practically independent of the cooling velocity and occurred very quickly with a pronounced heat effect.

Scheil¹⁸ attributed the spontaneous formation of martensite from austenite to the mechanical instability of austenite rather than to internal strains. On applying tensile stress to wires of austenitic nickel steels he found that the tensile strains raised the transformation 10 degrees Cent., but only when the grain size was equal to the cross section of the wire.

Bain¹⁹ found that a stable equilibrium condition of three phases, ferrite, austenite and carbide, was found to exist in a steel containing

¹⁵Wever and Möller, *Archiv. für das Eisenhüttenwesen*, Vol. 5, 1931, p. 215-18.

Regler, *Mitteilungen der Technischen Versuchsanstalten in Wien*, Vol. 20, 1931, p. 43-58.

¹⁶Steinberg, *Archiv für das Eisenhüttenw.*, Vol. 5, 1931-32, p. 383-5.

Steinberg and Zuzin, *Vestnik Metallopromishlennosti*, Vol. 12, 1932, p. 2-6.

¹⁷Wever and Engel, *Stahl und Eisen*, Vol. 50, 1930, p. 1308-11.

¹⁸Scheil, *Zeitschrift für Anorganische und Allgemeine Chemie*, Vol. 207, 1932, p. 21-40.

¹⁹Bain, *Transactions, American Institute of Mining and Metallurgical Engineers, Iron and Steel Division*, Vol. 100, 1932, p. 13-46.

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6.2 per cent manganese with 1.20 per cent carbon. This condition ultimately resulted after long heating at a proper temperature whether the original structure was austenitic or pearlitic. He found that "white martensite" was converted into a coarse ferrite carbide aggregation thousands of times faster than was austenite of the same composition and hence concluded that martensite cannot be an intermediate state in the austenite-pearlite reaction.

Hanemann and Wiester²⁰ found that austenite prepared by quenching high carbon steels in Wood's metal was extraordinarily stable up to about 250 degrees Cent., (480 degrees Fahr.), decomposing above this temperature to alpha iron and cementite. The beginning of the formation of martensite from austenite was stated to be independent of the velocity of further cooling. The temperature of the beginning of martensite formation increased with increasing carbon content, being 80 degrees Cent. for a 1.69 per cent carbon steel and about 240 degrees Cent. (465 degrees Fahr.) for a 0.83 per cent carbon steel. The martensite separation continued to low temperatures (—252 degrees Cent.) but even at this temperature some austenite still remained.

Hanemann and Schröder²¹ suggested that the name "perfusion" be given to the type of diffusion by which ferrite separates from austenite. It was assumed that certain preferred crystal directions were selected for diffusion and the external form taken by the segregate ferrite was determined by these crystallographic directions.

From a crystallographic investigation of two microscopic sections with a common edge and standing perpendicular to one another, Hanemann²² found that the martensite needles lie on octahedron and cube surfaces belonging together as in the Widmanstätten structure.

A study of the effect of low temperatures on chromium-nickel and nickel-silicon steels was made by Luerssen and Greene.²³ They found that in many cases the physical properties of these alloys could be greatly improved by cooling the quenched specimens to solid carbon dioxide temperature (—80 degrees Cent.) before tempering.

Mehl, Barrett and Smith²⁴ also investigated the Widmanstätten structure of steel and concluded that both the lattice type of the

²⁰Hanemann and Wiester, *Archiv für das Eisenhüttenwesen*, Vol. 5, 1932, p. 377-82.

²¹Hanemann and Schröder, *Zeitschrift für Metallkunde*, Vol. 23, 1931, p. 269-73.

²²Hanemann, *Archiv für das Eisenhüttenwesen*, Vol. 5, 1932, p. 625-26.

²³Luerssen and Greene, *TRANSACTIONS, American Society for Steel Treating*, Vol. 19, 1931-32, p. 501-544.

²⁴Mehl, Barrett and Smith, *American Institute of Mining and Metallurgical Engineers Contribution*, 37, 1933.

parent solid solution and the lattice type of the precipitate were important in determining the position and orientation of the precipitate. By measuring a large number of martensite needles they concluded that in all carbon steels the plane in austenite delineated by the martensitic structure is the (111), the mechanism being essentially that inherent in the gamma-alpha change in pure iron. Later, Mehl and Smith²⁵ arrived at the same conclusion with experiments on high purity iron.

Wiester²⁶ quenched a 1.7 per cent carbon steel in a metal bath at 100 degrees Cent., held it in a heater at this temperature while polishing and etching and then took motion pictures of the surface under the microscope as it cooled to room temperature. During the cooling, martensite needles were formed in the austenite grains, a whole "block" of needles usually forming simultaneously.

Esser, Eilender and Spenle,²⁷ found that the quenching velocity at which A_r' disappeared decreased up to 0.9 per cent carbon and then increased again. At this critical cooling velocity the temperature for the beginning of austenite transformation dropped continually from 650 degrees Cent. (1200 degrees Fahr.) at zero per cent carbon to 75 degrees Cent at 1.8 per cent carbon.

EXPERIMENTAL WORK

In the previous investigations of the effect of stress on decomposition of austenite by liquid air immersion a very important factor appears to have been largely overlooked. This factor is the irregularity of stress distribution caused by the ends of cylinders and the edges or corners of cubes or slabs. It is obvious that the ideal shape to use is the sphere. The reason that this shape has been so little used is, no doubt, that perfect spheres, particularly of the harder metals, are quite difficult to make. In spite of these attendant difficulties, it was thought worth while to make the following study on the effect of liquid air immersion on austenitized steel balls.

The steels selected for the investigation were a high carbon-chromium die steel and a typical ball-bearing steel of the following compositions:

²⁵Mehl and Smith, *Metals Technology*, Jan. 1934, Technical Publication No. 521, p. 1-7.

²⁶Wiester, *Zeitschrift für Metallkunde*, Vol. 24, 1932, p. 276-77.

²⁷Esser, Eilender and Spenle, *Archiv für das Eisenhüttenwesen*, Vol. 6, 1933, p. 389-93.

Steel	Type	Per Cent				
		C	Mn	Si	Cr	W
1	High C-Cr	2.14	0.26	1.14	10.37	0.61
2	Ball Bearing	1.00	0.28	0.26	0.74

Steel No. 2 was in the form of ball bearings of $\frac{3}{4}$ -inch diameter, the microstructure being shown in Fig. 5. This martensitic

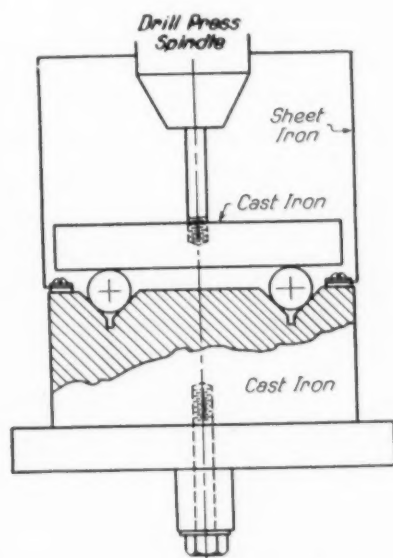


Fig. 1—Sketch of Ball Lapping Device.

structure is similar to a martensite with coarse needles except that it forms from a fine-grained austenite. The microstructure of steel No. 1, as received, is shown in Fig. 4.

The high carbon-chromium balls were hot-forged from a $\frac{5}{8}$ -inch diameter rod in a specially constructed die. In order to grind the specimens perfectly round, the lapping device shown in Fig. 1 was constructed.

The correct quenching temperature for the high carbon-chromium steel was determined by the following experiment. Six cylindrical specimens of exactly the same size were cut from the $\frac{5}{8}$ -inch rod. These were heated for ten minutes at different temperatures—1050, 1100, 1125, 1150, 1175 and 1200 degrees Cent. (1920, 2010, 2055, 2100, 2150, 2190 degrees Fahr.) respectively, and quenched in oil. A carbon resistor furnace of the plate type was used, so little decarburization could take place. Density and magnetic measurements were then made, as in Table I.

Table I
The Effect of Quenching Temperature on
Specimens of High Carbon Chrome Steel

Quenching Temp. Degrees Cent	Density	Magnetic Attraction Grams	Hardness Firth Diamond
1050	7.0785	0.086	768
1100	7.1358	0.064	486
1125	7.1561	0.022	392
1150	7.1551	0.014	382
1175	7.1534	0.013	376
1200	7.1540	0.014	369

In making the specific gravity determinations, ethylene dibromide was used instead of the customary water. This liquid was found to be very satisfactory: its density is about twice that of water, its viscosity is low and it is not very volatile at ordinary temperatures.

The ethylene dibromide appeared to be somewhat impure, so it was necessary to determine its density before using. The densities of this liquid for various temperatures were found to be:

Temperature, Degrees Cent.	Density
26	1.9869
28	1.9828
30	1.9789
32	1.9749
34	1.9709

These specimens were suspended from a fine copper wire, and correction was made afterwards for the volume of wire immersed. No correction was considered necessary for the capillary effect on the wire.

The magnetic attraction was determined by means of an analytical balance and a bar magnet. The magnet, of $\frac{5}{8}$ -inch round, was mounted in a vertical position over the pan of the balance and a V-notch of copper clamped to the pan made it possible to place all specimens in the same position under the pole of the magnet. All six specimens were weighed under the magnet without moving the magnet. The magnet was then removed and the true weight of the specimen determined. The difference in the two weighings was considered to be a measure of the magnetism.

Hardness determinations were made by grinding a considerable portion from one end of the specimen, the same amount being ground from each specimen. Five determinations were then made on each polished surface.

It appears to be impossible to make this steel completely non-magnetic. The melting point of the steel was not determined, but it is believed to be in the vicinity of 1200 degrees Cent. (2190 degrees Fahr.)

Table I shows that the maximum austenitic content was obtained somewhere between 1125 and 1200 degrees Cent. (2060-2190 degrees Fahr.) as evidenced by the fact that the density, magnetic attraction and Firth diamond hardness remained practically constant.

The balls of high carbon-chromium steel were austenitized by heating in borax glass to about 1150 degrees Cent. (2100 degrees Fahr.) followed by quenching in oil. The furnace was of the granular resistance type and the temperature was measured by means of an optical pyrometer, previously calibrated.

Density measurements were then made on three of the austenitized balls as well as on a $\frac{3}{4}$ -inch diameter ball of steel No. 2 which had been given the same heat treatment as the high carbon-chromium balls. Each of three balls (two of steel No. 1 and one of steel No. 2) was placed in the center of a spherical glass flask four inches in diameter and the flask filled with sil-o-cel. The junction of a calibrated copper-constantan thermocouple was placed at the center of another flask.

The flasks were immersed to the necks in liquid oxygen and temperature readings taken until the temperature of the liquid was approached. The flasks were then removed and permitted to return to room temperature. The cooling and heating curves are shown in Fig. 2. Density determinations were then made as before immersion.

An insulated high carbon-chromium ball was immersed directly in liquid oxygen for ten minutes and then removed and immediately put into the sil-o-cel around the liquid oxygen container so as to return slowly to room temperature. The differences before and after immersion are given in Tables II and III.

The microstructures for all of the specimens are shown in Figs. 4 to 11.

Since the cooling to the temperature of liquid oxygen and the return to normal temperature took place over a period of more than two hours, Fig. 2, the thermal stresses must have been very slight. In spite of these small stresses, a great deal of martensite was formed by the refrigeration in every case, Figs. 8 to 11. This martensite has the characteristic white needle structure with the mesial rib.

Table III shows that in the case of the completely austenitized

Table II
Effect on Density of Immersing Insulated Steel
Balls in Liquid Oxygen

Specimen No.	Type Steel	Quenching Temp. Degrees Cent.	Density		
			Before	After	Decrease
1	High C-Cr	1150	7.160	7.051	0.109
2	High C-Cr	1180	7.158	7.097	0.061
3	Low Cr Ball Bearing	1150	7.170	7.083	0.087
4*	High C-Cr	1150	7.160	7.044	0.116

*Uninsulated. Immersed 10 minutes and slowly returned to room temperature.

balls, the hardness after quenching is practically uniform over the cross sectional area. To obtain these hardness readings, as well as the micrographs, the balls were mounted in Wood's metal with half of the ball projecting. The projecting half was then ground off on an alundum wheel under a constant stream of cooling water and the section polished in the usual manner. Duplicate specimens were necessary, of course, in the case of the balls which were to be refrigerated. The hardness determinations of the "edge" were of necessity made about one millimeter from the outside surface of the spheres.

In the case of the ball bearing steel containing 1 per cent carbon, 0.74 per chromium, where the structure on quenching was largely martensitic, Fig. 8, the edge was harder than the center by 31 points. It will also be observed that the high carbon-chromium specimen No. 4, which was originally uniformly hard, upon direct immersion in liquid oxygen became definitely harder at the edge than at the center. Both of these cases indicate that tensile stress aids the formation of martensite.

In no case did the low temperature cause the decomposition of all of the austenite. Even in the case of the ball bearing steel there appeared to be some retained austenite after the liquid oxygen treatment (Fig. 11).

Table III
Effect on Hardness of Immersing Insulated
Steel Balls in Liquid Oxygen
 (Average of 10 Readings)

Spec. No.	Type Steel	Firth D.H. Before Imm.			Firth D.H. After Imm.			Average Increase
		Edge	Center	Mean	Edge	Center	Mean	
1	High C-Cr	384	382	383	814	816	815	432
2	High C-Cr	368	382	375	604	615	610	235
3	Low Cr Ball Bearing	814	783	798	909	899	904	106
4*	High C Cr	384	382	383	807	772	787	404

*Uninsulated. Immersed 10 minutes and slowly returned to room temperature.

The difference in the quenching temperature of specimens 1 and 2 (30 degrees Cent.) caused a great difference in the stability of the austenite as shown by density and hardness determinations. The specimen quenched from the higher temperature, Fig. 7, had a much smaller grain size than the other specimen, Fig. 6, probably because of a difference in the time of holding at the quenching temperature. In this case the greater stability of the austenite formed by quenching from the higher temperature was probably caused by the greater amount of dissolved carbide.

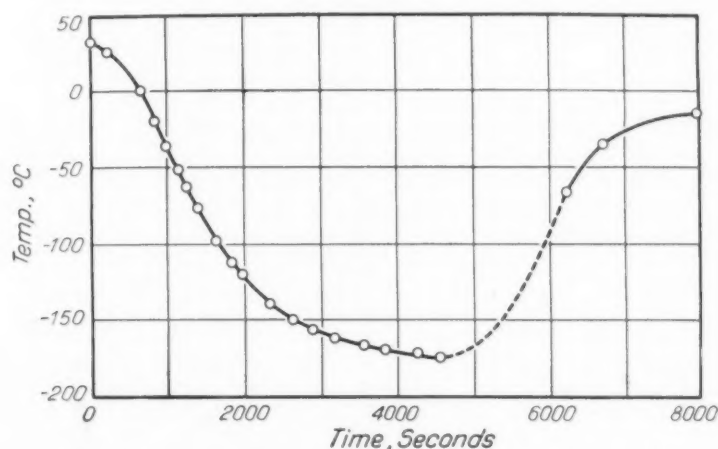


Fig. 2—Cooling and Heating Curve for Insulated Steel Balls Immersed in Liquid Oxygen.

As an additional experiment a thin piece of the high carbon-chromium steel, tapering in thickness from about 0.01 inch to a knife edge, was austenitized by quenching in oil from 1150 degrees Cent. It was then immersed for one-half minute in liquid oxygen. The density before immersion was 7.151 and after immersion 7.074. The hardness increased from a Vicker's diamond hardness of about 390 to about 790, a 10-kilogram load being used. The microstructures are shown in Figs. 13 and 14. The structure of the original austenite shows an unusually large number of twins. Just why more twinning should exist with this thin specimen than with the larger specimens is a matter for speculation.

In a thin blade about 0.002 inch thick (Fig. 13 at $\times 1000$) the thermal stresses arising on liquid air immersion must be extremely slight, and yet a considerable amount of martensite was formed.

In order to reduce thermal stresses to even smaller values, a thin specimen of the high carbon-chromium steel, about 0.01 inch

thick was insulated with sil-o-cel in the center of the 4-inch diameter flask. This was then immersed in liquid oxygen for an hour and a quarter. It will be observed that this was the time required for the similarly insulated steel balls to reach the temperature of liquid oxygen, Fig. 2. The flask was then removed and allowed to return to room temperature.

The microstructure after immersion showed a considerable amount of martensite (Fig. 15). The hardness increased from 390 to 770 Vicker's diamond.

MARTENSITE FORMATION AS OBSERVED BY THE MOTION PICTURE CAMERA

Another indication that thermal stresses are not essential to the formation of martensite from austenite was found by an experiment similar to that carried out by Wiester.²⁸ Instead of a 1.7 per cent carbon steel, however, it was found simpler to use a plain white cast iron, chill cast in about one-half inch section. The analysis of the iron used was carbon 3.31 per cent, manganese 0.39 per cent and silicon 0.07 per cent. The original microstructure is shown in Fig. 17.

The heater used for maintaining the specimen at about 100 degrees Cent. is shown in Fig. 3. A small coil of nichrome wire is placed in close proximity to the tapered hole which holds the specimen. The thermometer well may be unscrewed in case it is desired to make hardness determinations. The heater is so assembled that there is but little chance of any moisture finding its way into the heating element during the polishing. A wood block is screwed to the top of the heater for ease in handling. The thermometer was calibrated by a base metal thermocouple welded to the surface of a specimen placed in the heater during conditions of normal heating and normal cooling (current turned completely off). It was found desirable to use a small transformer (110-6v) and to place a rheostat in series with the heating element.

The specimen of white cast iron was heated near its melting point in borax glass for about an hour, quenched in Wood's metal at about 130 degrees Cent. and immediately transferred to the heater operating at 100 degrees Cent.

Keeping the surface of the specimen away at about 100 degrees Cent., it was roughly polished on sand paper resting on an electrically

²⁸Wiester, *Zeit. für Metallkunde*, Vol. 24, 1932, p. 276-77.

heated plate with the final polishing accomplished by boiling water and rouge on a rotating disk covered with wool felt. This disk was heated by means of a small Bunsen burner placed underneath. Etching was done by a hot 5 per cent solution of nitric acid in amyl alcohol, after which the surface was again slightly buffed to remove the tarnish.

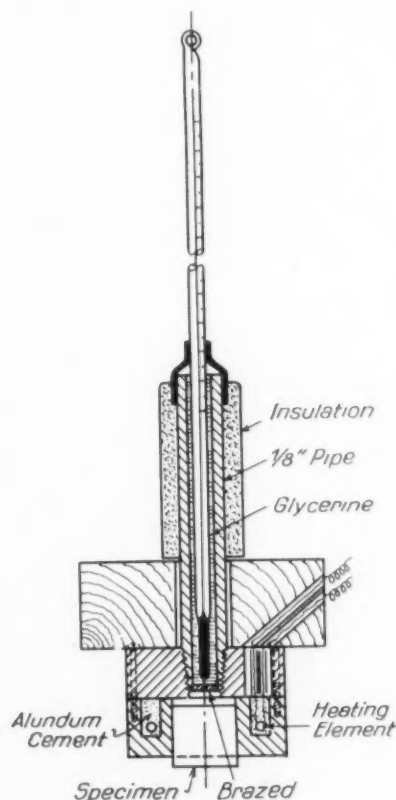


Fig. 3—Sketch of Specimen Heater.

Usually a small area of almost complete austenite was found on the surface of the various specimens. One of these areas, at 100 degrees Cent., is shown in Fig. 18. The same area, after cooling to room temperature, is shown in Fig. 19. Though the current to the heater was completely turned off, the cooling was very slow because of the large mass of the heater, and about one-half hour elapsed before any needles were observed to form and over one and one-half hours before room temperature was reached. The first needles appeared at about 50 degrees Cent., but needles continued to form to room temperature.

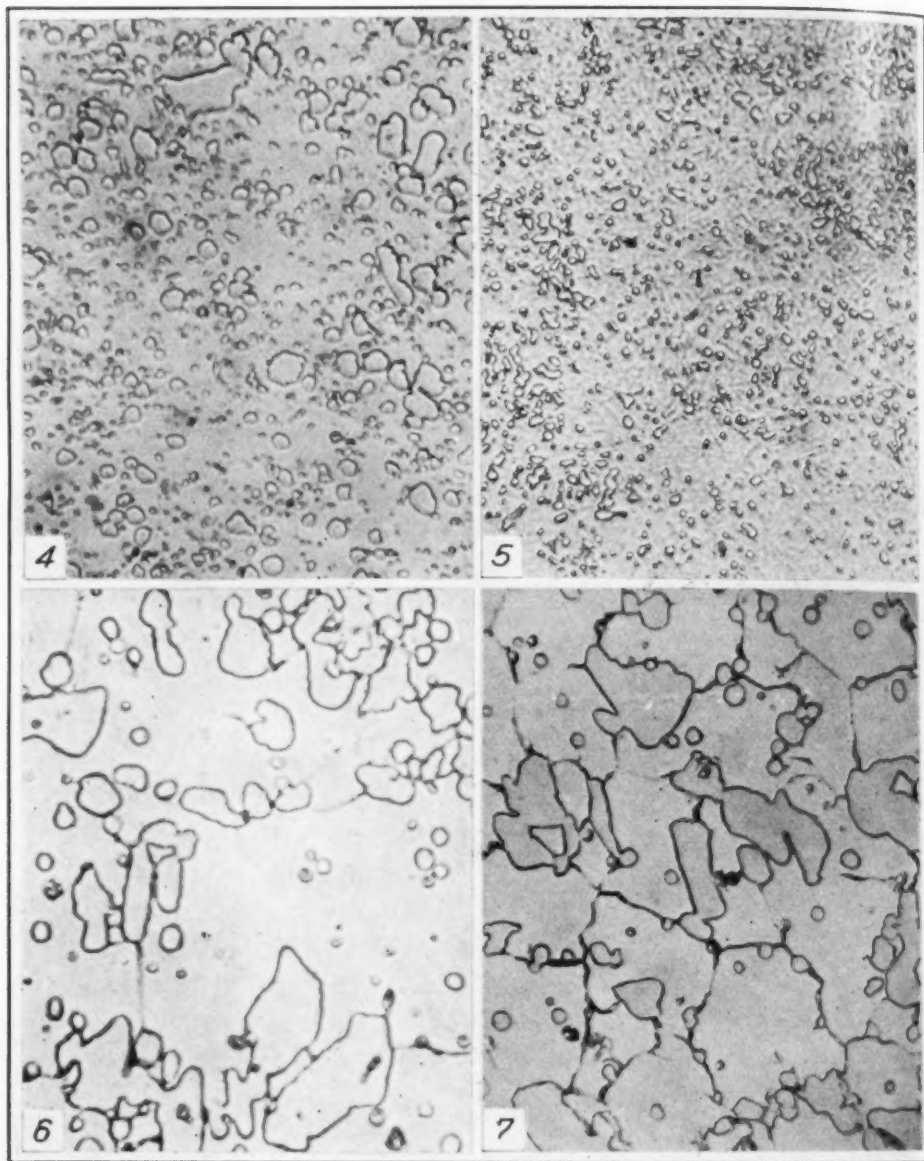


Fig. 4—High Carbon-Chromium Steel, As-Received, Showing Granular Pearlite. $\times 1000$.
 Fig. 5—Low Chromium Ball Bearing Steel, As-Received, Showing Carbide Plus Commercial Martensite. $\times 1000$.
 Fig. 6—Specimen 1 and 4. High Carbon-Chromium Ball Steel Quenched in Oil from 1150 Degrees Cent., Showing Austenite Plus Carbide. Firth Diamond Hardness, Edge 384, Center 382. Density 7.16. $\times 1000$.
 Fig. 7—Specimen 2. High Carbon-Chromium Ball Steel Quenched in Oil From 1180 Degrees Cent., Showing Austenite Plus Carbide. Firth Diamond Hardness, Edge 368, Center 382. Density 7.128. $\times 1000$.

The speed of formation of each needle was entirely too rapid to be followed by the eye. Usually several needles in more or less close proximity seemed to form at the same instant. In many cases

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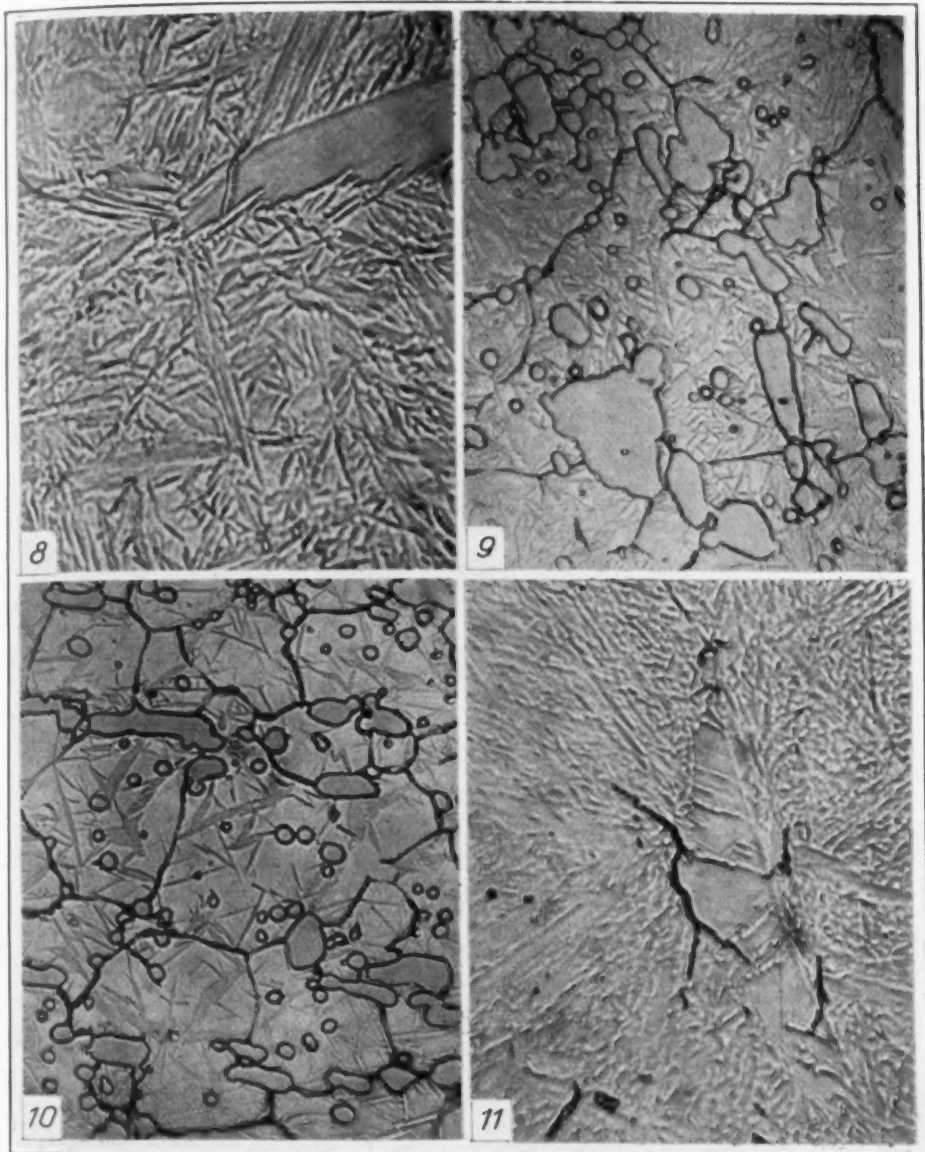


Fig. 8—Specimen 3. Low Chromium Ball Bearing Steel Quenched in Oil from 1150 Degrees Cent., Showing Martensite Plus Austenite. Firth Diamond Hardness, Edge 814, Center 783. Density 7.17. $\times 1000$.

Fig. 9—Specimen 1. High Carbon-Chromium Ball Steel Quenched from 1150 Degrees Cent. Insulated, Immersed in Liquid Oxygen, Showing Austenite Plus Carbide Plus Martensite. Firth Diamond Hardness, Edge 814, Center 816. Density 7.051. $\times 1000$.

Fig. 10—Specimen 2. High Carbon-Chromium Ball Steel Quenched in Oil from 1180 Degrees Cent., Insulated, Immersed in Liquid Oxygen, Showing Austenite Plus Carbide Plus Martensite. Firth Diamond Hardness, Edge 604, Center 615. Density 7.097. $\times 1000$.

Fig. 11—Specimen 3. Low Chromium Ball Bearing Steel Quenched in Oil from 1150 Degrees Cent., Insulated, Immersed in Liquid Oxygen, Showing Austenite Plus Martensite. Firth Diamond Hardness, Edge 909, Center 899. Density 7.083. $\times 1000$.

the "slipping" appeared to extend across a whole austenite grain. That these needles are really martensite may be noticed from their

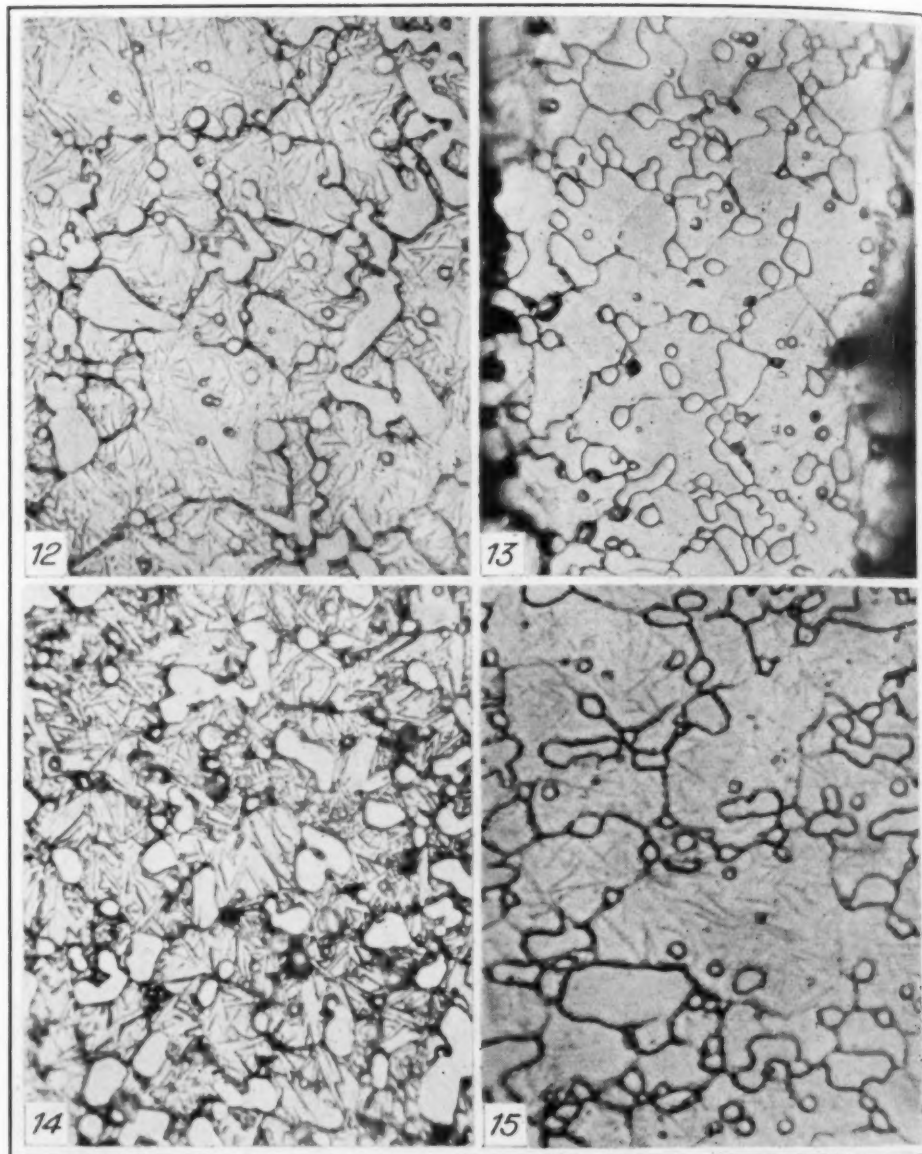


Fig. 12—Specimen 4. High Carbon-Chromium Ball Steel Quenched in Oil from 1150 Degrees Cent., Not Insulated, Immersed 10 Minutes in Liquid Oxygen, Showing Austenite Plus Carbide Plus Martensite. Firth Diamond Hardness, Edge 807, Center 772. Density 7.044. $\times 1000$.

Fig. 13—Thin Blade (0.002 Inch) of High Carbon-Chromium Steel Quenched in Oil from 1150 Degrees Cent., Showing Austenite Plus Carbide. Vickers Diamond Hardness 390. Density 7.151. $\times 1000$.

Fig. 14—Thin Blade of High Carbon-Chromium Steel Quenched in Oil from 1150 Degrees Cent., Immersed $\frac{1}{2}$ Minute in Liquid Oxygen, Showing Austenite Plus Carbide Plus Martensite. Vickers Diamond Hardness 790. Density 7.074. $\times 1000$.

Fig. 15—Thin Blade of High Carbon-Chromium Steel Quenched in Oil from 1150 Degrees Cent., Insulated, Immersed in Liquid Oxygen, Showing Austenite Plus Carbide Plus Martensite. Vickers Diamond Hardness 770. $\times 1000$.

appearance after repolishing and etching, Fig. 20. The characteristic zig-zag structure and the mid-rib are clearly shown. It is unlikely

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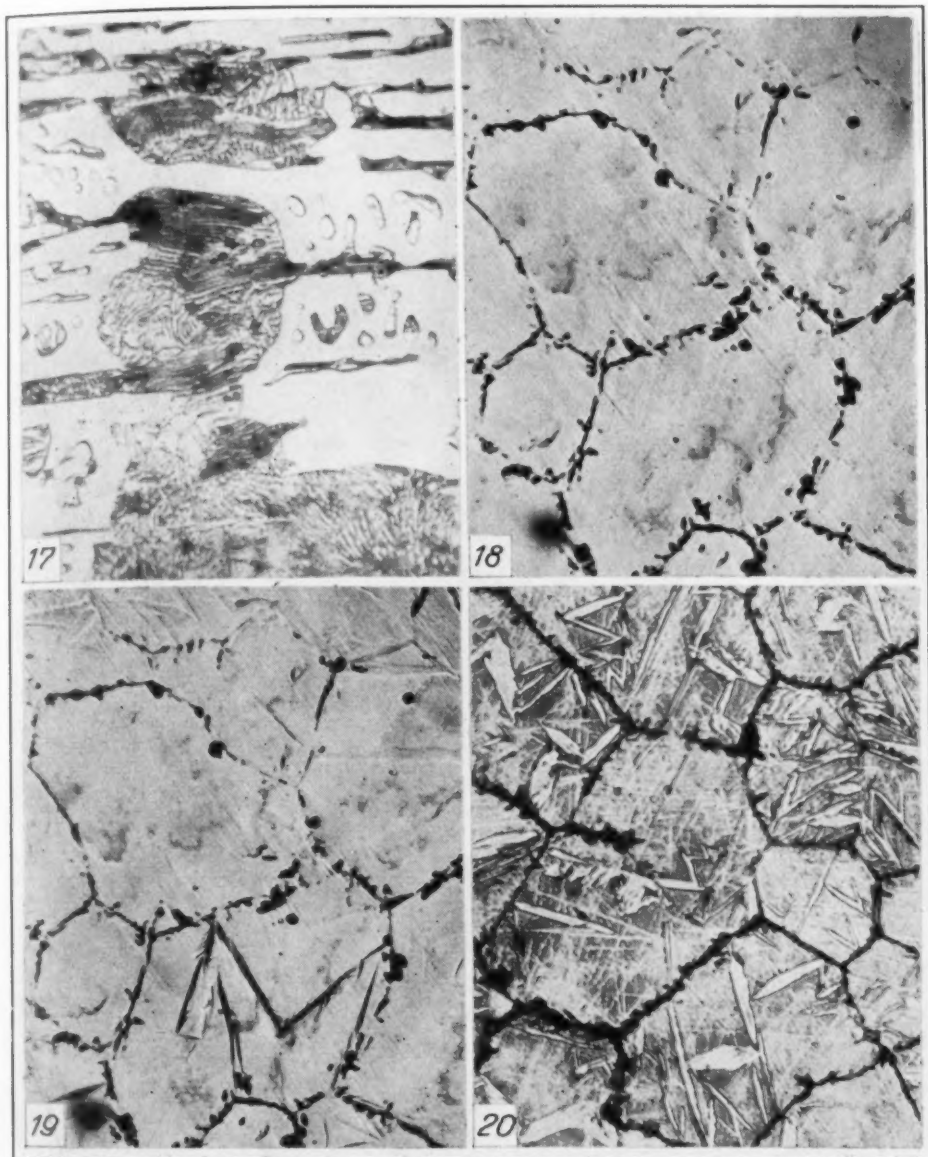


Fig. 17—White Cast Iron, as Cast, Showing Carbide Plus Pearlite. $\times 1000$.

Fig. 18—Austenitic Area in White Cast Iron Quenched from Melting Point in Wood's Metal at 130 Degrees Cent., and Held Above 100 Degrees Cent. Firth Diamond Hardness 361. $\times 200$.

Fig. 19—Same Area After Cooling to Room Temperature, Showing Austenite Plus Martensite. Firth Diamond Hardness 568. $\times 200$.

Fig. 20—Same Specimen After Cooling, Repolishing and Etching Showing Austenite Plus Martensite. $\times 200$.

that thermal stresses of any great magnitude could be set up during such a slow cooling as here occurred, and yet a considerable amount of martensite formed and the hardness increased from 361 at 100 degrees Cent. to 568 Firth diamond at room temperature.

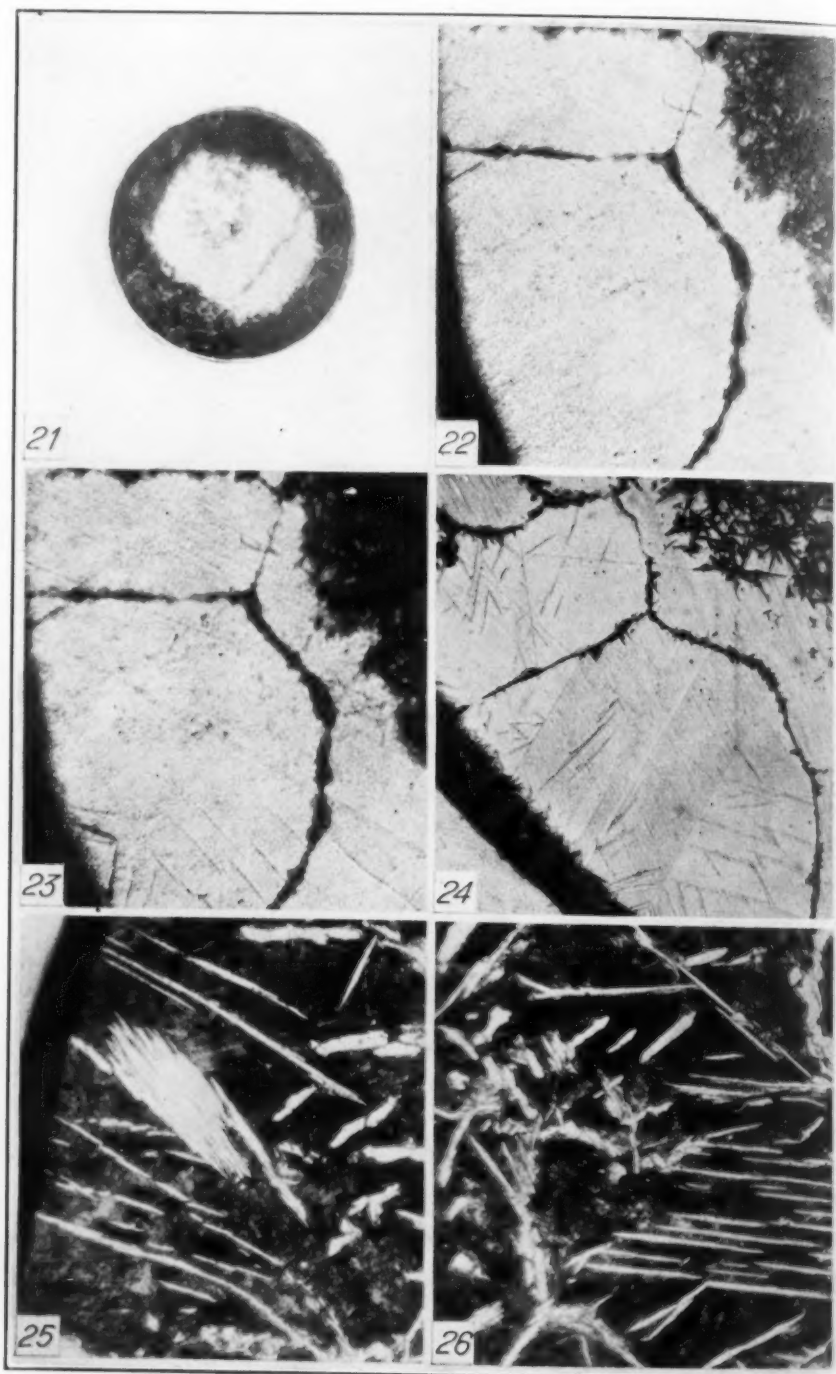


Fig. 21—Armco Wire Carburized 8 Hours at 1130 Degrees Cent., Quenched in Wood's Metal at 130 Degrees Cent. Showing Austenite Case, Martensite Core. $\times 15$.

Fig. 22—Austenite Case of Carburized Armco Wire at 100 Degrees Cent. $\times 100$.

Fig. 23—Same Case After Cooling to Room Temperature. $\times 100$.

Fig. 24—Same Case After Repolishing and Etching. $\times 100$.

Fig. 25—Case After Reheating and Slow Cooling. Carbon About 2.0 Per Cent. $\times 200$.

Fig. 26—Core After Reheating and Slow Cooling. Carbon About 1.6 Per Cent. $\times 200$.

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In order to get a better idea of the speed of formation of the needles, a motion picture was taken of the reaction. An ordinary motion picture machine was attached to the microscope in place of the usual camera. It was so adjusted that when the specimen was in visual focus in the side tube it would also be in focus on the film. Thus, the side tube could be pushed in occasionally during the taking of the pictures and the focus checked.

Even with the aid of these pictures, however, it was impossible to judge the speed of formation of each needle. On two successive pictures, taken at a speed faster than $\frac{1}{5}$ second per picture, the first frame showed no trace of a needle while the next showed the needle completely formed. This confirms the experiments by Wiester who noted that martensite needles formed faster than $\frac{1}{20}$ of a second.

Recently, Knight and Helmut Müller-Stock²⁹ observed the formation of martensite from several quenched steels when cooled to temperatures below room temperature. Microscopic examination showed the needles to form similar to the raising of a ridge by a mole, only much faster.

In order to prove conclusively that austenite can be retained in a pure iron-carbon alloy by quenching to just above the A_r'' , a $\frac{1}{8}$ -inch wire of Armco iron was pack carburized 8 hours at 1130 degrees Cent. (2065 degrees Fahr.). The specimen was then quenched in Wood's metal at 130 degrees Cent. and transferred to the heater as before. A tapered plug had been fitted to the heater and in this plug was a tapered hole to fit the wire specimen. As soon as the specimen was inserted in the heater it was cut off flush with the plug holder and prepared for the microscope as previously described.

On examination at 100 degrees Cent., the carburized wire was found to have an austenitic case and a martensitic core, Fig. 21. The structure of the case under higher magnification is shown in Fig. 22. This particular specimen was cut off with a high speed alundum disk and hence the martensite needles in the core were tempered somewhat and etched dark. This proved to be an advantage since the new martensite which formed on cooling could be readily distinguished from the tempered martensite (troostite) already present.

The structure after cooling to room temperature is shown in Fig. 23. The first needles were observed at about 41 degrees Cent., but some needles continued to form all the way down to room temper-

²⁹Knight and Helmut Müller-Stock, American Institute of Mining and Metallurgical Engineers Technical Publication No. 537, Feb. 1934, p. 1-7.

ature. After repolishing and etching, the surface appeared as shown in Fig. 24.

In order to estimate the carbon content, the specimen was reheated to 1130 degrees Cent. in a graphite crucible and cooled in the crucible. The surface and center then appeared as in Figs. 25 and 26. An estimation of the carbon content by means of the planimeter gave 2.0 per cent for Fig. 25 and 1.6 per cent for Fig. 26. A chemical analysis of the whole specimen gave a carbon content of 1.86 per cent.

SUMMARY

In this investigation evidence has been found that:

Thermal stresses are not essential to the formation of martensite from austenite.

The transformation of austenite to martensite in liquid air is initiated by the instability of the austenite at that temperature rather than by stress. Tensile stress, however, does tend to facilitate the formation of martensite.

The stability of retained austenite is increased by an increase in quenching temperature.

Austenite can be retained in a severely carburized pure iron by quenching to a temperature of about 100 degrees Cent. and holding at that temperature. A motion picture of the austenite—martensite transformation has been produced showing that the speed of formation of martensite needles is exceedingly great and that usually a large cluster of needles is formed at the same instant.

ACKNOWLEDGMENT

The writers wish to thank the various members of the Department of Metallography, University of Minnesota, and in addition Dr. A. T. Henrici, T. P. Hughes, F. Teal, Dr. H. Styri of the SKF Industries and the Air Reduction Company (Minneapolis Branch).

TORSIONAL MODULI VARIATIONS OF SPRING MATERIALS WITH TEMPERATURE

BY JOSEPH W. LUDEWIG

Abstract

The behavior of springs at elevated temperatures involves knowledge of the values of the modulus of rigidity, and proportional limits of materials at such temperatures. A summary of previous work upon the variation of the modulus of rigidity with temperature, and upon the behavior of springs at elevated temperatures is made.

The torsional moduli of a number of materials are plotted against temperature and in accordance with the procedure as outlined in this paper, the following results were obtained:

(1) Materials maintaining highest absolute value of the modulus of rigidity up to 450 degrees Fahr. are, high speed steel, stainless (cutlery) steel, Konel alloy.

(2) Material maintaining highest absolute value of the modulus of rigidity up to 985 degrees Fahr. is, high speed steel.

(3) Material showing lowest absolute value of the modulus of rigidity at all temperatures is, carbon spring steel.

(4) Remaining material tested was silchrome steel.

A discussion of the factors found in spring formulae and behavior under temperature variations is included in the paper.

INTRODUCTION

THE operation of spring materials over temperature ranges of from 0 to 1000 degrees Fahr. is taking place daily in certain instruments and mechanisms employed both in research enterprises and in industry. Besides being subjected to this very unusual operating range of temperature, some of these spring materials are subjected to the action of corroding and oxidizing atmospheres. Such atmospheres may contain CO, CO₂, N₂, H₂O, O₂, and numerous other gases in mixtures of varying amounts of each.

The author, Joseph W. Ludewig is a lieutenant in the U. S. Navy. This paper was submitted in partial fulfillment of the requirements for the degree of master of science, Carnegie Institute of Technology. Manuscript received Feb. 8, 1934.

Added to the combination of stress, variable temperatures and corrosion factors, there is another vital factor, namely, spring surge. This is due to vibration or surging, causing closing and opening of helical spring coils, particularly at the end coils. This is additional to that produced by the normal closing of the spring due to the cam, valve or other agency of action, and is produced at certain engine speeds giving rise to increased deflections, with attendant abnormal stresses and decrease of expected fatigue life.

These factors are taken into consideration to some degree for commercial springs, but the safe operation of springs at elevated temperatures, under corroding influences and under conditions of surge, has not been perfected.* The best static and fatigue properties of materials so far developed are used in spring design and the maximum energy that can be absorbed by a spring within its working range is thus found to be a criterion of its usefulness. However, the static and fatigue properties of various materials are not adequately determined, and it is the purpose of this paper to investigate one of these properties.

In a series of articles by J. K. Wood (1),² on spring design, certain valuable criteria have been evolved and demonstrated to be basic elements of design. These are the "spring criterion and material index." The "spring criterion" includes all dimensions of the spring and depends essentially upon the elastic property of the material. The "material index" has been defined as the ratio of $\frac{\pi S}{G}$ where S is the maximum safe fiber stress in pounds per square inch, and G is the torsional modulus in pounds per square inch. A definite relation between these two elements of design has been advanced and proven. It is stated that for a spring designed by the ordinary deflection formula, the spring criterion should not exceed the material index, or in symbols:

$$(\text{Spring Criterion}) \quad \frac{h - H}{HC^2} < \frac{\pi S}{G} \quad (\text{Material Index})$$

where h = maximum length of spring in inches D = mean diameter of spring
 H = solid length of spring in inches d = diameter of wire
 C = spring index = $\frac{D}{d}$ S and G are as noted above.

*The opinions and assertions contained herein are the private ones of the writer, and are not to be construed as official or reflecting the views of the Navy Department or the naval service at large.

²The figures appearing in parentheses refer to the bibliography appended.

The present work involves the study of the torsional modulus (as noted in the "material index") variations with temperature of a number of spring materials. These materials include a group of steels in use and of problematical value. One material has been furnished through the courtesy of C. R. Austin, of the Westinghouse Research Laboratories and consists of one of the high temperature precipitation hardening alloys recently developed there.

A discussion of the influence of various factors affecting the accuracy of load deflection formulae, commonly used in spring design, when applied to cylindrical helical springs of circular cross section where these springs are at elevated temperatures, is added to this paper.

REVIEW OF LITERATURE

The determination of the torsional modulus of various materials and the variation of this property with temperature, have been investigated by numerous authorities.

W. P. Wood (2), using the deflection method upon a formed spring has determined the modulus for a hard drawn steel, two tempered steels, and three nonferrous metals. The spring indexes $\frac{D}{d}$ used upon the samples were of the order of three, six, and nine, and the usual deflection formula, $G = \frac{8PD^3N}{Fd^4}$, applied. This formula with its constant of eight will apply and give quite accurate determinations of G as long as the spring index is greater than three. However, for springs of index of three or less, the increased linear shearing stress adjacent to the axis of the spring at the wire surface rises rapidly, and this usual formula, with constant eight, may not be used for such types of springs. The value of 11,450,000 is given for G as an average of values of the three steels at ordinary temperatures.

F. P. Zimmerli, W. P. Wood, and G. D. Wilson (3), have investigated the effect of temperature upon the torsional modulus of spring materials, and the important results as summarized are:

1. As temperature increases from 75 to 850 degrees Fahr., there is a general downward trend in the moduli.
2. In the temperature range of -100 to 75 degrees Fahr., alloy steels and nonferrous metals show no regular change in moduli. Carbon steels show a regular change in moduli through this range.

3. The modulus of stainless steel (18-8) is lower at all temperatures than that of other steels.

4. Monel metal shows great superiority at all temperatures to phosphor bronze and brass.

5. Cold work causes an apparent increase in the modulus, this effect reaching a maximum around 180 degrees Fahr. An explanation of this effect embodies the idea that the "trapped stresses" in the cold-worked metal neutralize part of the applied stresses, thus causing the modulus to appear higher than it really is.

A reproduction of some of the curves of this work is included for comparison, with the results of the author.

Recent investigations have been carried out and actual determinations made of the temperature coefficients of the modulus of rigidity, of aircraft instrument diaphragm and spring materials. Brombacher and Melton (4), have determined the modulus variation in the temperature range -20 to 50 degrees Cent. for three alloy steels, three high carbon steels and a number of nonferrous materials. This temperature coefficient is defined by:

$$m = \frac{1}{G_0} \frac{dG}{dT}$$

where m = temperature coefficient.
 G_0 = modulus of rigidity at 0 degrees Cent.
 G = modulus of rigidity at T degrees Cent.

A dynamic method with utilization of the torsion pendulum was used and the modulus computed from the standard formula for period, namely:

$$P^2 = \frac{8\pi LI}{Gr^4}$$

An interesting deduction was gathered from this paper, in that it was stated that for a high carbon, high chromium steel, the temperature coefficient of the modulus has a decided change for a tempering temperature of 500-600 degrees Cent. It is known that the carbide precipitation and agglomeration taking place in these steels at such temperatures, cause loss of corrosion resistance and strength, but the proven effect upon the modulus of rigidity is interesting. The steel mentioned has an analysis: carbon 0.81 per cent, manganese 0.129 per cent, chromium 13.2 per cent.

The aforementioned work has been continued by Keulegan and Houseman (5). The report of this work includes values of the temperature coefficient of thirty alloys and metals in the range of -50

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to 50 degrees Cent. The temperature coefficient of Poisson's ratio³ and significances in the differences in the two coefficients for a given material are discussed.

The temperature coefficients of the modulus of rigidity of a group of thirteen steels have been determined and all lie in the range of from 19 to 27×10^{-5} . Stainless (18-8) steels have temperature coefficients which are considerably higher than those of other steels. Accompanying these high temperature coefficients are high thermal coefficients of expansion in the stainless (18-8) types. Of the pure metals tungsten was found to have the smallest temperature coefficient and also the smallest coefficient of thermal expansion. This is in accord with the Andrew's modification of Wortheim's law, which includes a corollary stating for pure metals, a small temperature coefficient of elasticity is associated with a small thermal expansion.

This investigation, although carried on at low temperatures, determined that the coefficients in all cases increase in absolute value with increasing temperature.

Iokebi and Sakai (6) give the results of tests on thirteen different metals for the effects of temperature on the modulus of rigidity and on the viscosity of solid metals. They point out Benton's deduction (*Physical Review*) that the lower the melting point of a material, the greater is the rate of decrease of rigidity with rise of temperature and also suggest that annealing causes diminution of rigidity. A summary of their deductions is as follows:

1. As the temperature rises, rigidity of metals decreases according to parabolic laws.
2. With metals of low melting points, diminution of rigidity is very rapid, tending to zero at the melting points. For metals of high melting points, initial diminution is very small, but, as the temperature goes higher diminution of rigidity is more and more rapid.
3. In ferro-magnetic substances, logarithmic decrement and also rigidity in a less degree, are affected by the magnetic transformation, in that these are decreased by that transformation.
4. For metals of high melting point, logarithmic decrement decreases at first, reaches a minimum and afterward increases rapidly. The logarithmic decrement is in symbols:

$$\lambda = \frac{bT}{4I} \quad \text{where } \begin{array}{l} T = \text{period of oscillation} \\ I = \text{moment of inertia.} \end{array}$$

³Poisson's Ratio is defined as the ratio of lateral to longitudinal deformation and is denoted by λ .

Mallock (7) has also shown that the more infusible the material, the less is the rigidity affected by temperature changes.

As regards the actual selection of springs for work at elevated temperatures, work at Massachusetts Institute of Technology has demonstrated the following:

1. For work up to 500 degrees Fahr., silcrome steel suffers less permanent sets during a succession of loadings. Other steels tested were stainless (cutlery), nitralloy and high speed. These other steels will give satisfactory results, however.

2. For use of temperatures between 500 and 1000 degrees Fahr., high speed steel suffers less permanent set than any other steels examined.

Fritzie, Sutton, and Porter (8) describe the results of tests upon five different types of spring materials and give a detailed account of the heat treatment given to the superior spring material. The results are summarized in the amounts of permanent deformation in their springs after exposure to 5000 stress cycles at 700 degrees Fahr. These are:

Type	Rockwell Before	Hard. After	Perm. Deformation Amount	Average Composition					
				C	Mn	Cr	Al	V	W
Carbon Steel	48-54	40-43	0.047	0.95	0.4				
Stainless	44-48	43-47	0.005	0.12	0.5	14.0			
Nitralloy	37-41	38-44	0.01	0.44	0.5	1.6			
Cr-V	36-40	36-37	0.05	0.52	0.7	0.9	0.8		
High Speed	51-53	51-53	None	0.65	0.4	3.9		0.17 1.0	18.0

The superior high speed steel was quenched from 2260 degrees Fahr., which is the usual quenching temperature necessary to secure maximum properties in this type of material.

Thum (9) recommends several steels to combat higher temperatures and suggests the use of a steel with maximum carbide concentration. Some of the carbide forming elements which are V, Cr, W, Mo, Ta, Mn, Be, and Ti (to a lesser degree) are to be found in the types mentioned as S.A.E. chromium-vanadium 6150, the S.A.E. 1360 and high speed steels. The high speed steel is stated to have been successfully used up to 850 degrees Fahr. with a correspondingly low fiber stress of 35,000 pounds per square inch, and a Rockwell hardness of 44 to 46.

Stewart (10) states that for springs the manganese content of the steel is higher than that of the usual steels. It is also stated that the chromium-vanadium and silico-manganese types of springs can be operated at higher stresses than carbon steels. The modulus ac-

cepted at 11,500,000 is stated to be entirely independent of hardness or heat treatment.

For a given steel the hardness is, in a way, a measure of the tensile strength and related torsional strength. Work by Professor Moore, at the University of Illinois, has indicated that the endurance limit is also increased with hardness. Stewart states that the stress to which a spring can be subjected within the elastic limit will vary with the hardness. An interesting curve of torsional stress plotted against repetitions of stress shows that an S.A.E. 1360 steel, of about 150,000 pounds torsional strength has the asymptotic type of curve at about 50,000 pounds for 1,100,000 repetitions.

J. K. Wood (1), has pointed out that in the material index $\left(\frac{\pi S}{G} \right)$, heat and chemical influences affect S more than G . Such factors as usual impurities, lack of complete physico-chemical equilibrium due to mechanical and heat treatment, frequency of operation as related to fatigue and shock, electroplating, and corrosion are considered. Regarding impurities and alloy additions, it is stated that an excess of sulphur with its corrective addition of manganese will decrease the torsional modulus slightly and increase the shock resistance; that nickel additions will increase the elastic limit, ultimate strength and torsional modulus as will chromium and vanadium. The properly selected mechanical and heat treatment will increase the elastic limit, ultimate strength and have a slightly increasing effect upon the torsional modulus.

Jasper (11) has pointed out that the question of design can be divided in two general considerations as follows:

1. A consideration of the static, elastic and fatigue properties of the materials, and
2. A consideration of the shape of the springs, together with the distribution of stresses developed in use for a given deformation. The elastic properties as the modulus of elasticity and modulus of rigidity properly combined with the appropriate section modulus of the shape, determine the stiffness or load deflection rate. Now the question of design of springs to be used at elevated or depressed temperatures involves a knowledge of the values of the moduli of rigidity and elasticity at such temperatures. The use of certain energy relations derived by Sutherland in the *Philosophical Magazine*, 1891, has been used and checked for determination of modulus variations by Jasper (12). One of these simple energy relations,

$W = \frac{1}{2E} (S_1^2 + S_2^2) = K$, can for static testing be shown to be of the form, $W = \frac{S_1^2}{2E} = K_1$. This is shown to actually exist up to 950 degrees Fahr., thus enabling a calculation of the modulus of elasticity. For the modulus of rigidity, a formula for variation of this factor with temperature takes the form of

$$\frac{F}{F_0} = 1 - \left(\frac{T}{T_m} \right)^2,$$

where F = modulus of shear at any temperature absolute.

F_0 = modulus of shear at 0 degrees absolute.

T = temperature absolute at which F is obtained.

T_m = temperature absolute at which the metal melts.

This parabolic relation was plotted with $\frac{F}{F_0}$ as ordinate and $\frac{T}{T_m}$ as abscissae for Fe, Pb, Cu, Al, Au, Sn, Zn, and Ag and actual points checked with the curve. For steel an average value of the melting point was used for establishing the curve.

In the symposium (13), a discussion of valve spring steels, by Boegehold and Johnson, is interesting for inquiry into the temperature effects up to 500 degrees Fahr. Bailey, Eickenson, Inglis and Pearson point out the desirable qualities of high temperature steels, and P. Chevenard points out the necessity of a low internal friction as inherent quality for a spring, intended to work with rapid oscillations. He states that the breakage of springs proceeds from an accumulation of the effects of hysteresis over a very large number of cycles.

THEORETICAL AND PRACTICAL CONSIDERATIONS OF INVESTIGATION OF VARIATION OF TORSIONAL MODULUS WITH TEMPERATURE

J. K. Wood (1) has quite clearly summarized the four methods of determination of the torsional modulus, and further discussion of these methods is found in the work of W. P. Wood (2) and Zimmerli, Wood, and Wilson (3). These four methods have been stated as:

1. The direct method
2. The deflection method
3. The torsional pendulum method
4. The elongation method.

The direct method involves the use of a torsional moment upon a straight sample and direct calculation of the modulus from the ratio of unit fiber stress to unit angular deformation. Thus:

$$G = S \div \frac{\theta}{L} = \frac{583 \text{ PRL}}{d^4 \theta}$$

where P = load in pounds
 R = lever arm in inches
 L = length of sample in inches
 d = diameter of sample in inches
 θ = angle of twist in degrees
 G = torsional modulus in pounds per square inch.

The deflection method involves the use of a helical spring under various degrees of loading and a measure of the consequent deflection of this spring. The deflection formula, derived from the previous formula, enables the determination of the modulus. It is stated in either of the forms:

$$G = \frac{8PD^3H}{d^3(h-H)} = \frac{8PD^3N}{d^4F}$$

where P = load in pounds
 D = pitch diameter of spring in inches
 H = solid length of spring in inches
 h = maximum length of spring in inches
 d = diameter of spring wire
 N = number of active coils = $\frac{H}{d}$
 F = deflection in inches.

The torsional pendulum method, used by a number of investigators as noted in the reference of previous work, involves the use of a sample wire pendulum, weighted and set in motion by a twisting force. The usual relation between moment of inertia, period of oscillation and physical dimensions of the pendulum, give the relation for the modulus as follows:

$$G = \frac{2\pi IL}{t^2 r^4}$$

where I = moment of inertia of system
 L = length of pendulum
 t = period of oscillation
 r = radius of sample wire.

The elongation method involves the measurement of the elastic modulus in tension and calculation of the torsional or rigidity modulus from the Poisson ratio relation. This is:

$$G = \frac{E}{2(1+\lambda)}$$

where E = modulus of elasticity
 λ = Poisson's ratio.

Certain observations by J. K. Wood (2), and Wood, Zimmerli and Wilson (3), as regards the deflection method and the inherent simplicity of the direct method have led the author to select this last named method for study of the torsional modulus. The deflection method is subject to two variable elements. The factor of eight in the formula has been noted to hold only for that type of spring in which the linear shearing stress adjacent to the axis of the spring at the wire surface is negligible. This, of course, occurs when the spring index $\left(\frac{D}{d}\right)$ is relatively large, and thus limits the experimental work.

The bending stresses present in a formed helical spring might also have a small effect upon the modulus.

Keulegan and Houseman (5), have noted that the torsional pendulum method has serious disadvantages, and also record some information of work done upon the thermal coefficient of Poisson's ratio. The work upon the Poisson's ratio coefficient is apparently meager, and precludes an attack of the problem of rigidity modulus variation from the standpoint of a solution of the elastic modulus variation in the elongation method.

The selection of specimens for the direct method of solution of the modulus is apparently similar to that made by Zimmerli, Wood and Wilson (3). A double hooked specimen of approximately ten inches gage length is held between two slotted shaft vises, one fixed, the other possible of rotation through the means of the light steel sheave and weights. The shaft vises were constructed of high speed steel (whose modulus was previously known to hold up quite well at elevated temperatures), and two pierced stainless steel circular disks over the face of each shaft vise, centered the specimens, and at the same time allowed free longitudinal movement of specimen. This last arrangement, for centering of specimens, allows freedom from bending stresses which might be induced by expansion of the specimens at elevated temperatures. The rotating shaft vise moves in a double ball bearing arrangement, the sheave, very carefully machined to an eight inch diameter, is fastened at a fixed length from the shaft vise, and measurement of angular deflection is made from movements

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of the music wire suspension which carries the twisting load. An adjustable slider upon this wire moves past a scale graduated in one hundredth inch divisions, and measurements to an accuracy of five thousandths inch are made easily with the aid of a small magnifying glass. These linear measurements are changed into angular measure by means of a simple conversion table. Calibration of the music wire for unit extension under various loads was made prior to the experiments and the amount of error in readings from this source shown to be negligible.

The amount of angular deflection in the shaft vises was also computed for various loads, and the error from this source shown to be negligible.

The question of the type of sample to be employed, that is, one of the tubular or one of the solid type, was given careful thought. The solid wire type was actually selected, because of lack of elaborate machining facilities necessary to the provision of carefully formed tubular specimens. Further research into the work of previous investigators, particularly that of Templin, and Moore (14), shows that comparative results from hollow and solid specimens will give little variation in values of the modulus of rigidity. However, as is commonly known, the tubular specimen is designated by these authors as the type to be used when determinations of shearing strength, elastic limit, and yield point are to be made.

The matter of temperature control presented the usual difficulties, and the apparatus finally selected consisted of a nichrome-wound furnace, constructed in such a manner as to give as small a temperature gradient as possible. This furnace was controlled by means of a rare metal couple, set against the winding and connected to a recording pyrometer. Into this furnace of eighteen inches length was placed a close fitting pure copper muffle of fourteen inches length, to further the non-gradient condition. At each end of this copper muffle was placed a small nichrome booster coil set into the face of transit disks. Each inner disk was backed up by a second disk at a distance of about one inch away toward the ends of the furnace, the air space between acting as additional insulation against heat losses. The shaft vises, of course, rotated within these disks and were given sufficient clearance to prevent the occurrence of any friction. With the above arrangement, the gradients over a 10-inch gage length were measured and found to be not more than two and one-half degrees Fahr. at any temperatures. Every run made was checked

were carried out with several factors in mind. A set of cyclic loadings was carried out upon each specimen over a range of temperatures from normal atmospheric to the highest temperature in the range of testing. The effects of elastic afterworking and permanent set had to be carefully guarded against, and the first provision was, of course, to remove all stress from the specimen during periods devoted to tem-

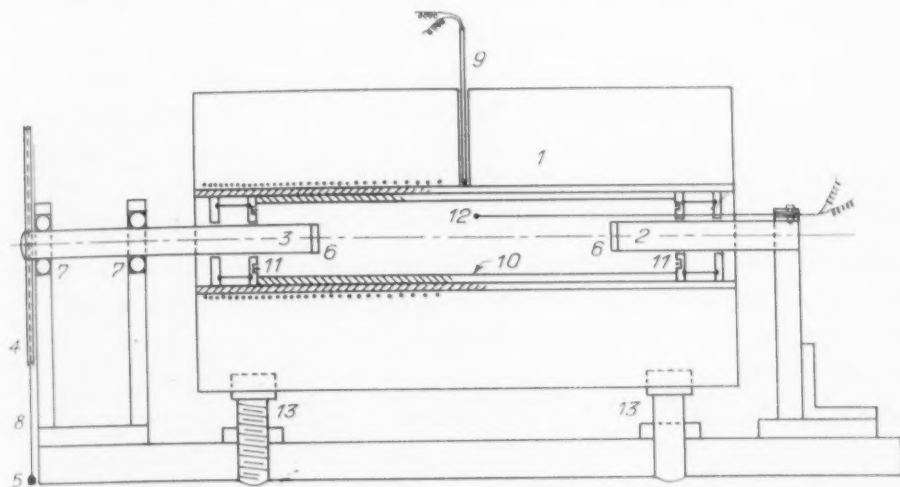


Fig. 2—Diagrammatic Sketch of Apparatus Used in this Investigation.

1. Nichrome-Wound Furnace; 2. Fixed Vise; 3. Rotating Vise; 4. Light Steel Sheave; 5. Weights; 6. Pierced Stainless Steel Disks; 7. Ball Bearings; 8. Music Wire Suspension; 9. Controlling Thermocouple; 10. Copper Muffle; 11. Booster Coils; 12. Gradient Measuring Couple; 13. Furnace Supports.

perature changes, and at all times when deflections were not actually being recorded. If elastic afterworking and permanent set occurred, then the stiffness of a spring at a certain temperature would no longer remain constant. Keulegan and Houseman (5), have pointed out that the stiffness depends, to a small degree, upon the deflection and duration of deflection. Keulegan and Houseman (5) further state that both elastic afterworking and permanent set are causes of actual increase in deflection with time, while load is maintained constant, but differ in that when the load is removed, the excess deflection is recovered only in the case of elastic afterworking. In the cyclic method, it only becomes necessary to note the difference between initial and final reading at the instant of completing the cycle, which, if negligible, proves that elastic afterworking is negligible. It is further evident, according to these authors, that this difference has also included the differential permanent set, and thus the single criterion is sufficient for both if the difference is negligible. Accordingly in each measurement made by the author, the initial loading of six-tenths pound

(placed upon the specimen both for an initial reference point and to keep the music wire straight, and from recoiling upon the sheave) was removed from the specimen during periods of temperature change, by means of the stay noted at the end of the sheave. Also a series of cyclic loadings were made upon each specimen at each temperature when equilibrium temperature conditions were attained. The loads were applied slowly, deflection reading taken at each increment of load, and then the specimen completely unloaded. The initial loading was removed from the specimen by manually rotating the steel sheave in reverse direction equivalent to the deflection which this initial load would have caused. Then this load was allowed to stress the specimen again, and a check made to see whether initial and final readings agreed. A number of cycles were performed before going to another temperature for testing purposes. To check against the use of a single specimen for a series of tests, numerous other specimens were tested at only one temperature and then removed from the apparatus. The checks were good. A matter of interest has been noted in the reports of J. K. Wood (1), in which he states that Guthrie gave interesting facts concerning fatigue, in which he showed the area of the hysteresis loop, i.e., the area between loading and unloading lines on a pressure deflection chart is a measure of the susceptibility of the material to fatigue. He stated that a material whose pressure deflection chart shows a relatively very small hysteresis loop will probably withstand fatigue fairly well. In the author's work, hysteresis was noted and recorded to a limited extent during the earlier progress of the experiment, prior to knowledge of the above reference. Some comparative hysteresis loops are included for information purposes.

A set of calibrated weights was made up for loading of specimens, and the stress range over which tests were made was, in most cases, from 6000 to 32,000 pounds per square inch. The loads and stresses are noted:

Load	
.6 pound	6,250 pounds per square inch
1.1 pounds	11,450 pounds per square inch
1.6 pounds	16,650 pounds per square inch
2.6 pounds	27,000 pounds per square inch
3.1 pounds	32,200 pounds per square inch

These stresses are calculated according to the usual formula:

$$S_r = \frac{16M_t}{\pi d^3} \quad \text{where} \quad \begin{array}{l} S_r = \text{shearing unit stress at distance } r \text{ from center} \\ M_t = \text{torsional resisting moment} \\ d = \text{diameter in inches.} \end{array}$$

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The modulus of elasticity has been shown by Sayre (15), to be of a curved nature dependent upon stress intensity. Considerable work upon the properties of spring steel in bending, with respect to hysteresis and overstressing, has been sponsored by the Research Committee on Mechanical Springs, of the A.S.M.E., under J. K. Wood, as chairman, and further contributions, by Sayre, to this work are found in the works (16), (17), (18). The curvature of the modulus of rigidity under different conditions of stress intensity was not seriously considered in the present investigation, due to lack of time and requisite accuracy for the investigation. An average modulus at each temperature is recorded. From readings taken, however, a rough consideration shows that Sayre's results are corroborated in this investigation for the torsional modulus of the spring materials. The materials under investigation are as follows:

Material	Chemical Analysis								
	C	Mn	S	P	Si	Cr	V	W	Ni
High Speed Steel	0.72	0.27	0.024	0.43	3.9	1.03	18.0
	(1) Preheat 1500 degrees Fahr., oil quench from 1800 degrees Fahr. Temper at 1050 degrees Fahr.								
	(2) Preheat 1500 degrees Fahr., oil quench from 1950 degrees Fahr. Temper at 1050 degrees Fahr.								
Stainless (Cutlery) Steel	0.38	0.27	0.02	0.02	0.21	14.07	0.22
	(1) Preheat 1250 degrees Fahr., oil quench from 1825 degrees Fahr. Temper at 900 degrees Fahr.								
	(2) Preheat 1250 degrees Fahr., oil quench from 1650 degrees Fahr. Temper at 900 degrees Fahr.								
Silchrome Steel	0.46	0.40	0.019	3.23	8.77
	(1) Preheat 1500 degrees Fahr., oil quench from 1800 degrees Fahr. Temper at 1200 degrees Fahr.								
Carbon Spring Steel	0.61	1.31	0.017	0.016	0.14
	(1) Oil quench from 1500 degrees Fahr. Temper at 560 degrees Fahr.								
	(2) Oil quench from 1525 degrees Fahr. Temper at 560 degrees Fahr.								
Konel Alloy No. 2948	Ni	Co	Fe	Ti	Cr	(1) Quench from 950 degrees Cent. Age harden at 650 degrees Cent. for 72 hours.			
	46	25	7.5	2.5	19				

The Rockwell hardnesses of the above steels were as noted below:

High speed steel1800 degrees Fahr. quench—Rockwell C49-52
High speed steel1950 degrees Fahr. quench—Rockwell C53-55
Stainless (Cutlery)1825 degrees Fahr. quench—Rockwell C46-50
Silchrome steel1800 degrees Fahr. quench—Rockwell C35-40
Carbon spring steel1525 degrees Fahr. quench—Rockwell C53-55
Konel Alloy No. 2948	..Age hardened at 650 degrees Cent. for 72 hours—Rockwell C40-41

RESULTS AND CONCLUSIONS

The results of calculations of the torsional moduli based upon the data gathered in these tests, is incorporated in a series of graphs, Fig. 3 to 13. These data are presented in Tables I to V.

The series of graphs Figs. 3 to 13 comprise: 1. A set of curves depicting the changes of torsional moduli with temperature for all

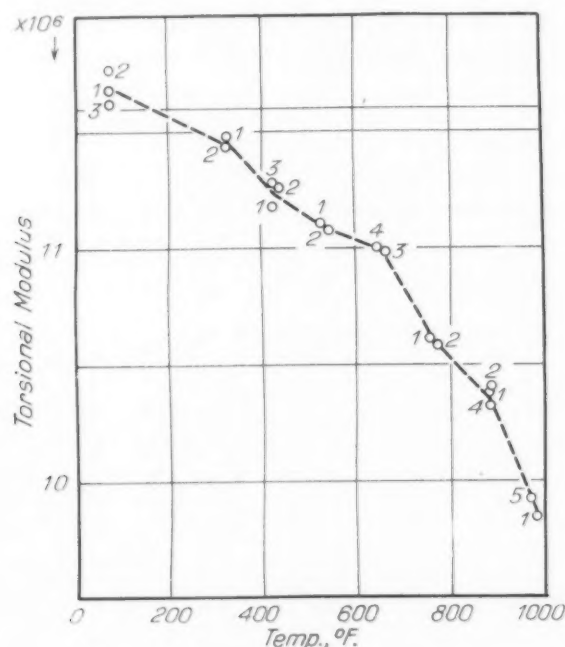


Fig. 3—Torsional Modulus Versus Temperature of High Speed Steel.

of the materials tested; 2. A group of individual curves for each material and indicating the plot of each modulus with its temperature; 3. A set of curves compiled from the work of previous investigators, and that of the author.

The group of individual curves show the close agreement between different specimens at the various temperatures of test. Each plotted point is the result of a series of from three to eight successive cyclic loadings over the range of 6000 to 32,000 pounds unit shear stress. The variation in temperature over the gage length during the time of taking readings was not more than $2\frac{1}{2}$ degrees Fahr., and usually about 1 degree Fahr. The individual curves have been plotted through the mean value of modulus where a group of plotted points in the near vicinity of each other resulted. The tests upon some of the materials, which showed a large increase in the

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Table I
Composite Data for High Speed Steel

Specimen	Twist	θ	Length	Temp. Degrees Fahr.	G
1	56.3	8.06	9.87	77	11,680,000
1	57.3	8.20	9.87	329	11,480,000
1	58.7	8.41	9.87	427	11,180,000
1	59.1	8.46	9.87	530	11,110,000
1	61.9	8.86	9.87	759	10,610,000
1	63.4	9.08	9.87	885	10,380,000
1	66.8	9.56	9.87	982	9,850,000
2	56.3	8.06	9.96	77	11,770,000
2	57.9	8.30	9.96	329	11,440,000
2	58.9	8.44	9.96	442	11,260,000
2	59.9	8.58	9.96	545	11,080,000
2	62.7	8.99	9.96	774	10,580,000
2	63.8	9.14	9.96	890	10,400,000
3	66.4	9.51	9.9	977	9,930,000
3	56.5	8.09	9.86	77	11,620,000
3	58.3	8.35	9.86	424	11,280,000
3	59.8	8.56	9.86	662	10,990,000
4	59.5	8.52	9.86	646	11,020,000
4	63.5	9.10	9.86	883	10,320,000

Table II
Composite Data for Cutlery Steel (CrS-5)

Specimen	Twist	θ	Length	Temp. Degrees Fahr.	G
1	56.1	8.03	9.85	79	11,700,000
1	57.1	8.18	9.85	336	11,480,000
1	58.5	8.38	9.85	430	11,200,000
1	59.2	8.48	9.85	538	11,070,000
1	61.1	8.75	9.85	664	10,720,000
1	63.8	9.14	9.85	761	10,290,000
2	56	8.02	9.9	79	11,760,000
2	57.7	8.27	9.9	350	11,410,000
2	58.6	8.40	9.9	455	11,220,000
2	61.8	8.85	9.9	662	10,790,000
2	67.2	9.64	9.9	888	9,800,000

Table III
Composite Data for Silchrome Steel

Specimen	Twist	θ	Length	Temp. Degrees Fahr.	G
1	57.7	8.26	9.92	77	11,460,000
2	57.7	8.26	9.95	78	11,500,000
3	57.7	8.26	9.86	78	11,400,000
3	59.7	8.55	9.86	340	11,010,000
3	60.7	8.70	9.86	455	10,810,000
3	64.9	9.29	9.86	689	10,130,000
3	67	9.59	9.86	824	9,820,000
4	61.7	8.84	9.86	586	10,650,000
4	66.4	9.51	9.86	817	9,890,000

absolute value of the temperature coefficient of modulus, have been confined to a lesser number of plotted points. However, for the two materials showing the least variation of modulus with temperature, namely, high speed steel and stainless (cutlery) steel, a con-

Table IV
Composite Data for Carbon Spring Steel

Specimen	Twist	θ	Length	Temp. Degrees Fahr.	G
1	61.1	8.75	9.87	77	10,780,000
1	63.1	9.04	9.87	360	10,420,000
1	67.2	9.63	9.87	482	9,780,000
2	60.9	8.72	9.86	77	10,800,000
2	64	9.16	9.86	388	10,290,000
3	65.1	9.32	9.9	394	10,130,000
3	69.8	9.99	9.9	500	9,450,000
4	60.9	8.72	9.88	77	10,800,000
4	66.7	9.55	9.88	426	9,890,000
5	60.4	8.65	9.84	77	10,850,000
5	66.3	9.49	9.84	421	9,890,000
5	68.7	9.85	9.84	518	9,540,000

Table V
Composite Data for Konel Alloy

Specimen	Twist	θ	Length	Temp. Degrees Fahr.	G
1	56.4	8.08	9.82	77	11,600,000
1	59.2	8.48	9.82	444	11,050,000
1	60.2	8.63	9.82	662	10,860,000
1	63.3	9.06	9.82	833	10,330,000
2	59.5	8.52	9.84	554	10,990,000
2	61.9	8.86	9.84	781	10,580,000
2	67.7	9.70	9.84	975	9,660,000

siderable number of tests have been run, and the plotted points found in close agreement. The small variations in heat treatment given to high speed steel, stainless, and carbon spring steel created no apparent differences in the results. However, small differences in hardness and constituents present at normal temperatures, due to this variation in heat treatment, should be noted. Keulegan and Houseman (5), have found that in the case of alloy steels, the effects of heat treatment upon the temperature coefficients are not very definite, that is, the differences found were of the same order as the errors of experimentation.

A consideration of the set of curves of all materials shows a general downward trend in the torsional modulus as temperature is increased above normal. It is to be regretted that measurements could not be made in the temperature range of 100 to 300 degrees Fahr. inclusive, but the apparatus available did not allow of accurate temperature control in this range. Due to the factor just mentioned, the best apparent solution seemed to be to connect the first modulus measurement above normal temperature by straight line with the modulus recorded for normal room temperature. The succeeding determinations at higher temperatures have been connected by

straight lines to their preceding plotted points. In addition to the increase in absolute value of the temperature coefficients, which is apparent from first glance at these curves, the parabolic resemblance

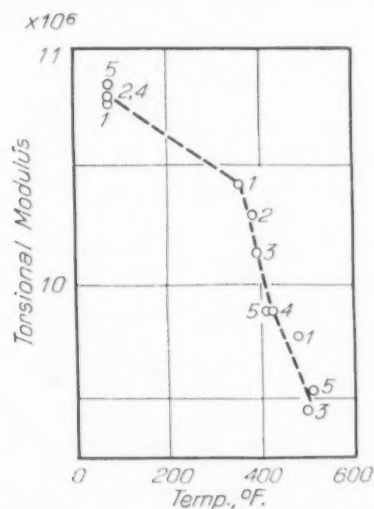


Fig. 4—Torsional Modulus Versus Temperature of Carbon Spring Steel Grade C, U. S. N. Spec. 47-5-4.

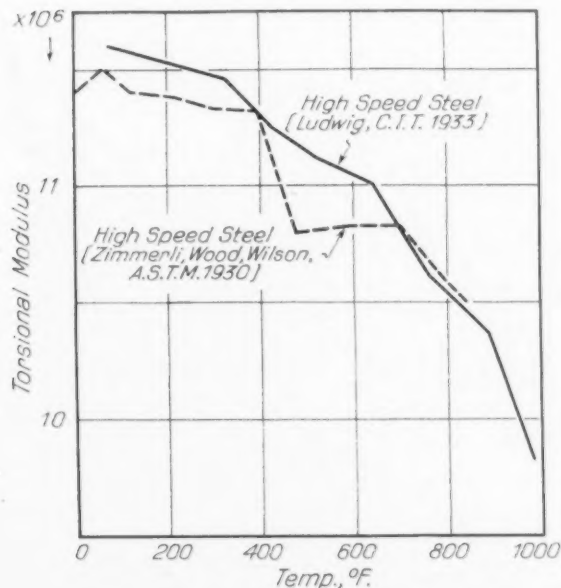


Fig. 5—Torsional Modulus Versus Temperature of High Speed Steel. Dotted Line (Zimmerli, Wood, Wilson) Solid Line (Ludwig).

is to be noted. Numerous authorities have shown this relationship to exist for the pure metals and also for some carbon steels. In a survey of the work of Sutherland, on the kinetic theory of solids,

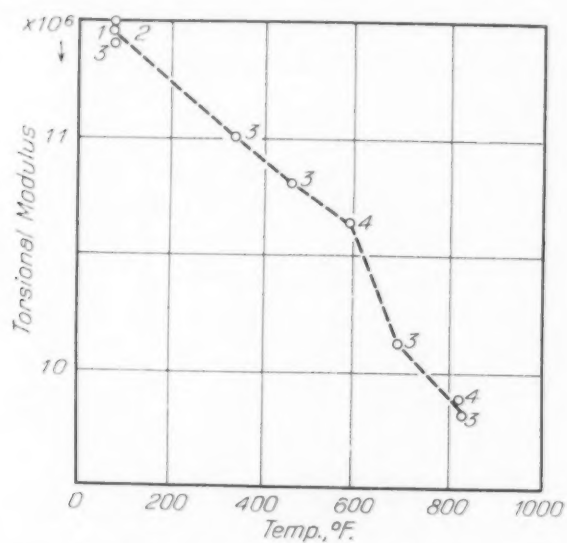


Fig. 6—Torsional Modulus Versus Temperature of Silchrome Steel.

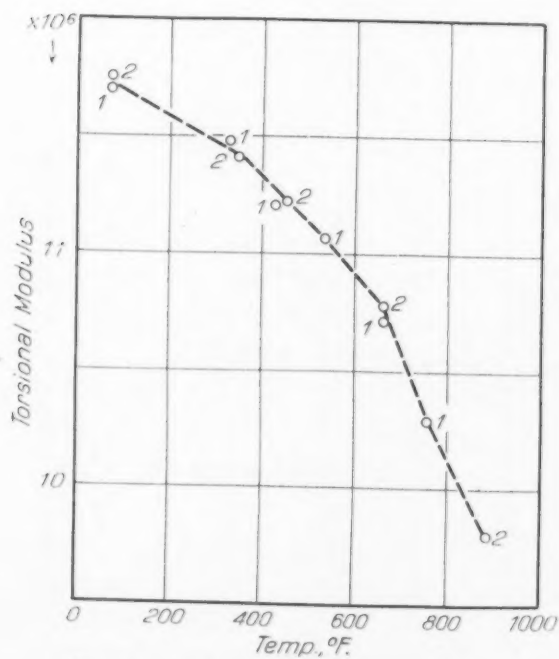


Fig. 7—Torsional Modulus Versus Temperature of Steel C.H.S.-5, U. S. N. Spec. 46-8-18.

Jasper (12) has shown a parabolic curve of the following form:

$$Y = 1 - x^2$$

The reproduction of this curve is shown in Fig. 14.

Likewise a series of curves by Iokebi and Sakai (6), for car-

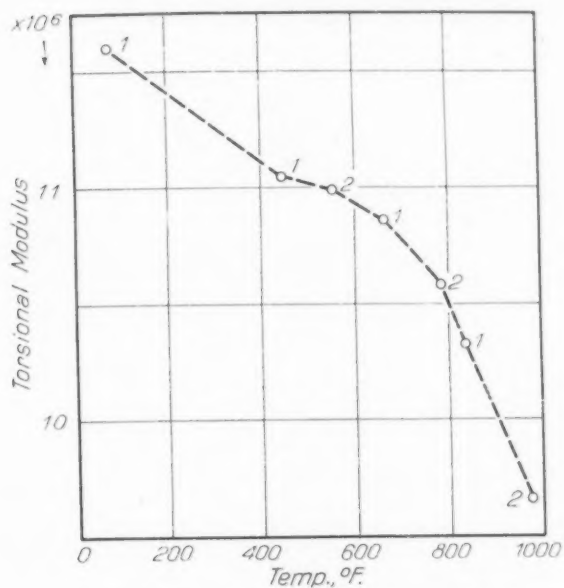


Fig. 8—Torsional Modulus Versus Temperature of Konel Alloy.

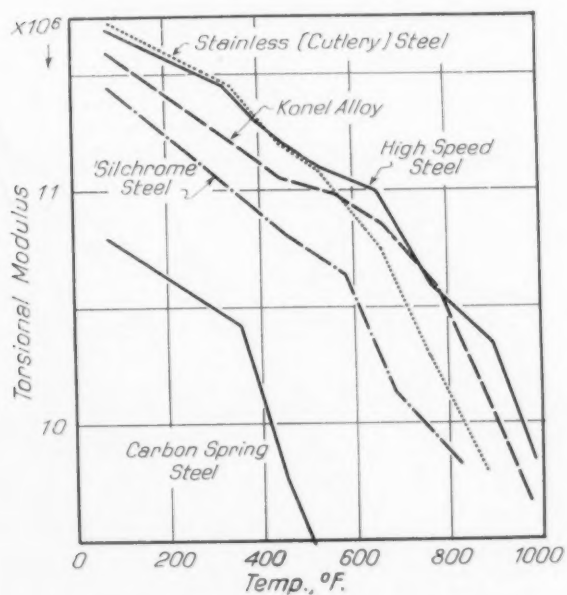


Fig. 9—Torsional Modulus Versus Temperature of Five Materials.

bon steels of variable carbon content show this parabolic relation. The reproduction of these curves is shown in Fig. 15.

SUMMARIZED RESULTS

In the light of the present investigation made upon the series

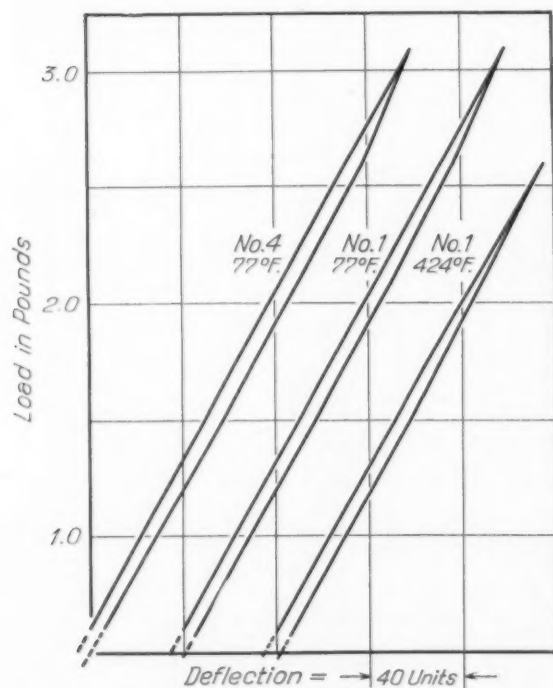


Fig. 10—Typical Hysteresis Curves of High Speed Steel.

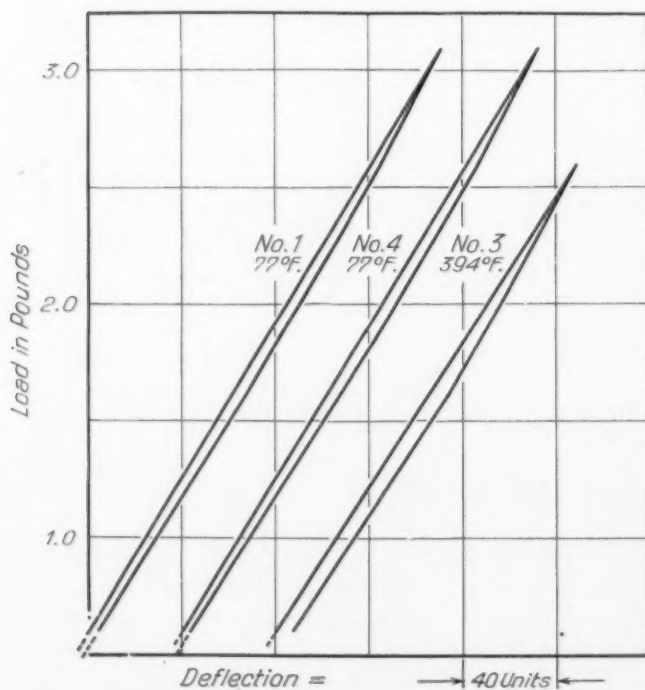


Fig. 11—Typical Hysteresis Curves of Carbon Spring Steel.

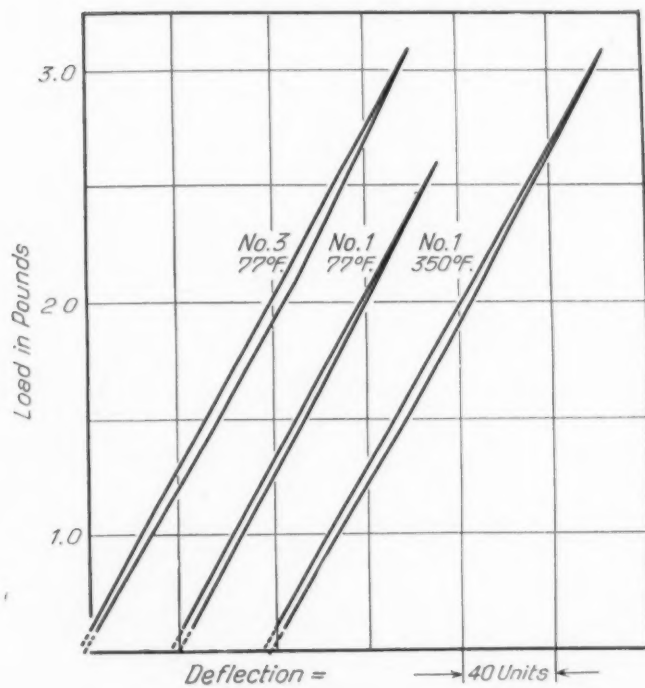


Fig. 12—Typical Hysteresis Curves of Stainless (Cutlery) Steel.

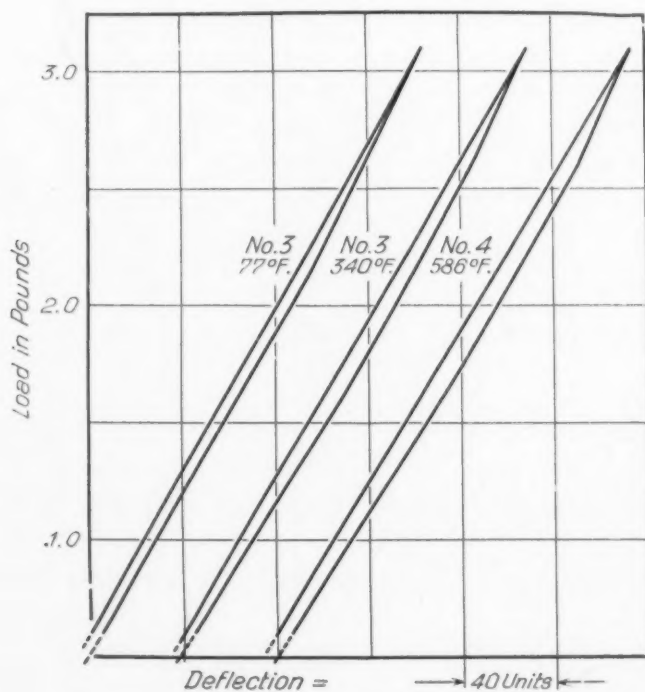


Fig. 13—Typical Hysteresis Curves of Silchrome Steel.

of materials outlined heretofore, these materials are classified as follows:

1. Materials which maintain the highest absolute value of the modulus of rigidity (lowest temperature coefficient of moduli) up

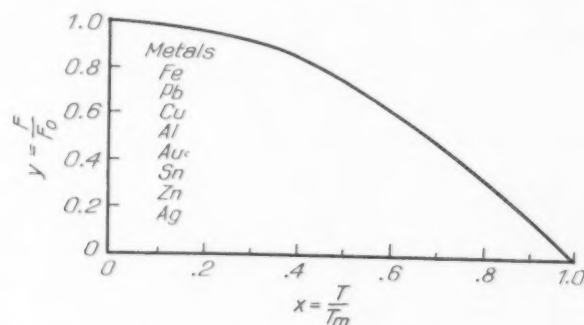


Fig. 14—Reproduction of Parabolic Curve of Sutherland.

to 450 degrees Fahr. are high speed steel, stainless (cutlery) steel and Konel alloy.

2. The material maintaining highest absolute value of the modulus of rigidity up to 985 degrees Fahr., high speed steel.

3. The material showing lowest absolute value of the modulus of rigidity at all temperatures, carbon spring steel.

It is also apparent that the most highly alloyed materials have been the types which showed the least variation of rigidity with temperature increases. The modulus variation of all materials resembles a series of parabolic types of curve relations. In the earlier part of this paper, a set of relations, stated by Wood (1), was brought out. The basic elements are repeated:

$$(\text{Spring Criterion}) \frac{h-H}{HC^2} < \frac{\pi S}{G} \quad (\text{Material Index})$$

The above condition has been shown to be obtained by equating the maximum allowable work applied to a spring to its resilience. The resilience, in turn, is the maximum amount of useful work recoverable after the removal of the load.

The elongation or depression of the spring as noted in the Spring Criterion is identified in the symbols by $\frac{h-H}{H}$. This factor can be computed according to the following relation:

$$\frac{h-H}{H} = \frac{P}{2G} \times \left(\frac{D}{d} \right)^2 \times \frac{D}{d^3}$$

where P = maximum operative load in ounces
 G = torsional modulus of the material
 D = mean diameter of the spring in inches
 d = diameter of the wire in inches
 H = solid length of the spring in inches
 h = maximum length of the spring in inches
 C = spring index = $\frac{D}{d}$

This formula is identified as the Deflection Formula by J. K. Wood, and it can be seen that it depends upon the elastic property

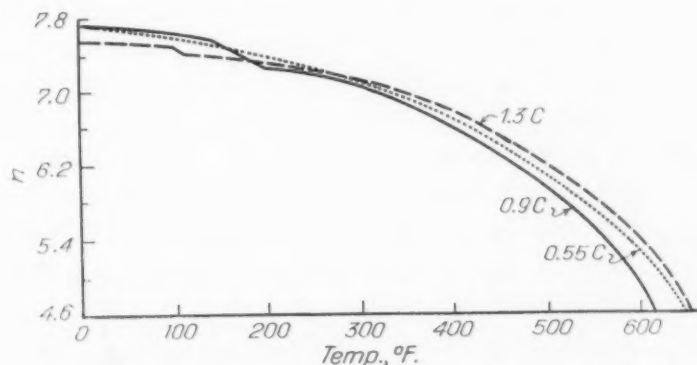


Fig. 15—Reproduction of Curves for Carbon Steels by Iokichi and Sakai.

of the material and includes all dimensions of the spring. The elongation or depression of the spring varies inversely as the torsional modulus and the square of the wire diameter, but directly with the cube of the spring index, and first power of the maximum operative load. Now the safe maximum working load has been defined according to the Capacity Formula as noted below:

$$P_e = \pi \times 2S \times \frac{d^3}{D}$$

where P_e = maximum safe working load in ounces

S = maximum safe fiber stress in pounds per square inch

The dependency of the maximum safe working load upon the strength and elastic limit of the material is to be noted.

SUMMARIZED CONCLUSIONS

The summarized conclusions from a close consideration of the above relations show the following:

1. A material should be used, maintaining as high an elastic limit as possible, and preferably a low value for the torsional modulus, in order to give the most serviceable spring.

2. Good spring practice necessitates the use of a Spring Index (C) value of not less than seven.

3. With the requirements of pressures and elongations known, the Deflection Formula will give a value of wire diameter necessary to use, but the Spring Criterion < Material Index. That is, no more work should be applied to a volume of wire by load P than that which the material is able to give out again as useful work, the internal energy being known as the resilience of the particular volume. J. K. Wood has stated that a spring made of any steel or phosphor bronze, with Spring Index greater than seven, under any condition of service, but not elongated nor depressed more than 50 per cent, is safe for the maximum load designed.

4. The important material properties to be known of a spring when in the state it will be as a finished spring, are its elastic limit and modulus. These vary with temperature. For rough work, a value of the torsional elastic limit is sometimes taken as about 50 to 60 per cent of the tension elastic limit.

The question of use of the torsional elastic limit, in order to determine whether the maximum safe fibre stress shall not be exceeded, is one of some doubt. Is not the endurance or fatigue limit of the material at the elevated temperature a more just criterion for limiting safe fiber stress? Jasper (11) has given a deduction wherein he states that the endurance limit stress is the strength property to use with appropriate method of determining the factor of safety, rather than the elastic limit. He further states that, in general, for spring steels, as temperature rises, the fatigue and static strength values decrease in a progressive manner. A number of correlations of endurance limits to proportional limits, yield points, ultimate strengths and Brinell hardnesses are given and the closest correlation noted for the relations to ultimate strengths and Brinell hardnesses. For a series of nineteen steels, the results of rotating beam methods give the ratio:

$$\frac{\text{Endurance Limit in Reversed Torsion}}{\text{Endurance Limit in Reversed Flexure}} = 0.53$$

The endurance limits are related to springs in that the stress range through which spring operation takes place from initial to

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maximum load, governs the maximum stress to which the spring can be subjected. Automotive valve springs operating in the range of 175 to 250 degrees Fahr. are usually designed so that the steel is subjected to a maximum stress of 75,000 pounds per square inch, with a stress range of 20,000 pounds per square inch. High carbon steel properly heat treated meets these demands, whereas a chromium-vanadium steel will operate over a stress range of 30,000 pounds per square inch, and at a maximum stress of 85,000 pounds per square inch. Should higher maximum stresses be desired, then the range will have to be decreased.

The most careful considerations in design will not prevent spring failures, however, if the processing of the spring is performed in a haphazard manner. The most serious apparent causes of spring failures are usually surface cracks, flaws, embrittlement by plating, lowering of the proportional limit by tinning or Japanning, presence of foreign elements as slag and inclusions, decarburization, segregation and heat treatment defects. The last is commonly found as improper heat treatment, that is, failure to secure the maximum proportional limit (or endurance limit) in the material. Again it is found by heat treatments given at the wrong time in the processing of the spring. It has been found that the best uniformity and durability are obtained by coiling springs with annealed wire, and heat treating after coiling to a spring temper. The processing by coiling heat treated wire and subsequently heating to relieve strains gives less uniformity and durability. A statement is given by Boegehold and Johnson (13), that one spring manufacturer gives an increased life of 5 to 1 for carbon steel springs, and 11 to 1 for chromium-vanadium springs for processing by the more beneficial method.

In general, heat treatments may retard or may accentuate the following changes taking place in materials under stress at elevated temperatures:

1. Grain growth
2. Coalescence of constituents in case of aggregates
3. Homogenization of heterogeneous solid solutions
4. Structural hardening of supersaturated solid solutions
5. Tempering phenomena in the case of alloys previously treated or cold-worked
6. Rate of approach to the limited spacing state between

adjacent atomic layers beyond which restoration does not take place completely upon removal of load. This is the proportional limit.

Hysteresis effects in materials and their relation to spring fatigue is given in Figs. 3 to 13. These are representative of the various materials. The area of such hysteresis loops is a measure of the materials susceptibility to fatigue.

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